RSC Advances

Supporting information

I_2-Promoted cross-dehydrogenative coupling of α-carbonyl aldehydes with alcohols for the synthesis of α-ketoesters

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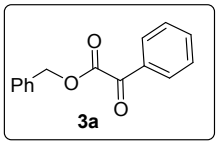
Experimental Section

General: IR spectra were recorded on a Bruker Tensor 37 (FTIR) spectrophotometer. ^{1}H NMR spectra were recorded on Bruker Avance 400 (400 MHz) spectrometer at 295 K in $CDCl_3$; chemical shifts (δ in ppm) and coupling constants (J in Hz) are reported in standard fashion with reference to either internal standard tetramethylsilane (TMS) (δ_H = 0.00 ppm) or $CHCl_3$ (δ_H = 7.25 ppm). ^{13}C NMR spectra were recorded on Bruker Avance 400 (100 MHz) spectrometer at RT in $CDCl_3$; chemical shifts (δ in ppm) are reported relative to $CHCl_3$ (δ_C = 77.00 ppm). In the ^{1}H-NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, m = multiplet and br s = broad singlet, sept = septet. The assignment of signals were confirmed by ^{1}H and ^{13}C spectral data. High-resolution mass spectra (HR-MS) were recorded on an Agilent 6538 UHD Q-TOF using multimode source.

All small scale reactions were carried out in 5 ml stoppered round bottom flask (RBF). Reactions were monitored by silica gel TLC plates, using a mixture of petroleum ether and ethyl acetate as eluents. All solvents were distilled prior use; petroleum ether with a boiling range of 60 to 80^{o}C, dichloromethane (DCM), ethyl acetate, purchased from locally available commercial sources were used. Alcohols were purchased from locally available commercial sources and few of them from Sigma Aldrich. α-Carbonyl aldehydes were prepared according to literature procedure.[1]

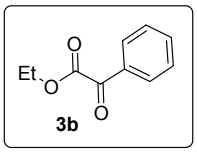
1**. General Procedure for the Synthesis of α-Ketoesters:** To a mixture of phenylglyoxal mono hydrate **2a** (1 mmol) and benzyl alcohol **1a** (1.2 mmol) in toluene (2 ml), iodine (1 equiv.) and K_2CO_3 (2 equiv.) were added and the resulting reaction mixture was stirred at room temperature for appropriate time until the starting materials were completely consumed. The products were purified by column chromatography on silica (petroleum ether/ethyl acetate). All the compounds were confirmed by FTIR, ^{1}H NMR, ^{13}C NMR and HR-MS Spectral analyses. Among 21 compounds, 8 (**3f-3h**, **3l**, **3m**, **3n**, **3p & 3r**) are unknown and 13 (**3a-3**e, **3i-3k**, **3o**, **3q**, **3s-3u**) are known. We gave FTIR, ^{1}H NMR & ^{13}C NMR spectral data for known compounds and FTIR, ^{1}H NMR, ^{13}C NMR & HR-MS spectral data for unknown compounds.

Spectral Data of all Compounds (3a-3u)



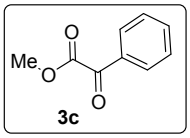
3a

Benzyl 2-oxo-2-phenylacetate (3a):[2] Colorless liquid (94%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 3065, 3047, 17323, 1649, 15956, 1451, 1293, 1170, 745, 677. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.93 (m, 2H), 7.55 (m, 1H), 7.35-7.45, (m, 4H), 7.32 (m, 3H), 5.37 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 186.0, 163.6, 134.9, 134.5, 132.3, 129.9, 128.8, 128.9, 128.5, 67.6.



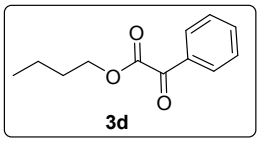
3b

Ethyl 2-oxo-2-phenylacetate (3b):[2] Colorless liquid (94%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2984, 1733, 1687, 1299, 1196, 1013, 674. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 8.01 (d, J = 7.3 Hz, 2H), 7.65 (m, 1H), 7.51 (m, 2H), 4.45 (q, J = 7.3 Hz, 2H), 1.42 (t, J = 7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 186.5, 163.9, 134.9, 132.5, 130, 128.9, 62.3, 14.1.

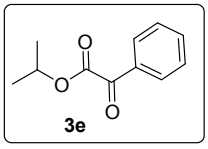


3c

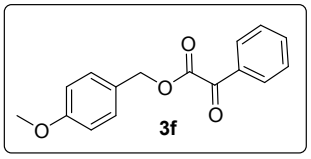
Methyl 2-oxo-2-phenylacetate (3c):[3] Colorless liquid (95%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2956, 1737, 1688, 1203, 1001, 750, 677. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 8.01 (d, J = 7.8 Hz, 2H), 7.66 (m, 1H), 7.51 (m, 2H), 3.98 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 186.1, 164.1, 135, 132.4, 130.1, 128.9, 52.8.



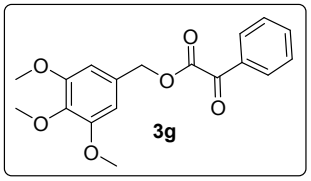
Butyl 2-oxo-2-phenylacetate (3d):[3] Colorless liquid (92%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2961, 2935, 1733, 1687, 1451, 1299, 1195, 984, 675. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 8 (m, 2H), 7.65 (m, 1H), 7.5 (m, 2H), 4.39 (t, J = 6.6 Hz, 2H), 1.76 (m, 2H), 1.44 (dq, J_a = 15 and J_b = 7.4 Hz, 2H), 0.96 (t, J = 7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 186.5, 164.0, 134.9, 132.5, 129.9, 128.9, 66.0, 30.4, 19.0, 13.6.



Isopropyl 2-oxo-2-phenylacetate (3e):[4] Light yellow oil (90%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2984, 2925, 1730, 1689, 1597, 1451, 1202, 1101, 986, 733, 672. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.98 (d, J = 7.8 Hz, 2H), 7.63 (m, 1H), 7.49 (m, 2H), 5.31 (sept, 1H), 1.39 (d, J = 6.4 Hz, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 186.7, 163.6, 134.8, 132.5, 129.9, 128.9, 128.8, 70.6, 21.7.

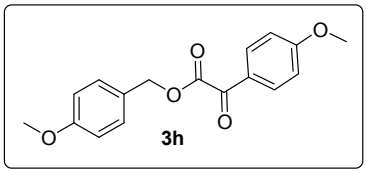


4-Methoxybenzyl 2-oxo-2-phenylacetate (3f): Colorless liquid (88%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 3004, 2960, 2838, 1732, 1684, 1611, 1514, 1248, 1167, 967, 737, 674. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.93 (m, 2H), 7.6 (m, 1H), 7.4 (m, 2H), 7.37 (d, J = 8.8 Hz, 2H), 6.89 (d, J = 8.8 Hz, 2H), 5.34 (s, 2H), 3.77 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 186.2, 163.8, 160.1, 134.9, 132.5, 130.6, 130.0, 128.9, 126.7, 114.1, 67.7, 55.3. HR-MS (ESI+) m/z calculated for $[C_{16}H_{14}NaO_4]^+$ = $[M+Na]^+$: 293.0784; found: 293.0767.

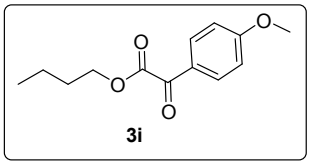


3,4,5-Trimethoxybenzyl 2-oxo-2-phenylacetate (3g): Colorless liquid (85%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2940, 1734, 1685, 1454, 1331, 1237, 1122, 968, 687. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.98 (d, J = 7.3 Hz, 2H), 7.66 (m, 1H), 7.5 (m, 2H), 6.68 (s, 2H), 5.35 (s, 2H), 3.87 (s, 6H), 3.86 (s, 3H). ^{13}C

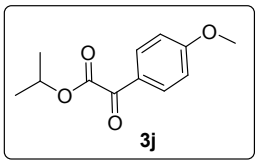
NMR ($CDCl_3$, 100 MHz): δ_C = 186.0, 163.6, 153.4, 135.0, 132.4, 130.0, 128.9, 105.8, 67.9, 60.9, 56.3, 56.1. HR-MS (ESI+) m/z calculated for $[C_{18}H_{18}NaO_6]^+$ = $[M+Na]^+$: 353.0996; found: 353.0985.



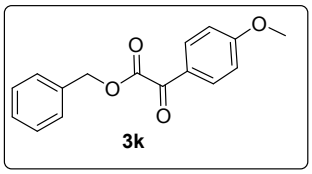
4-Methoxybenzyl 2-(4-methoxy phenyl)-2-oxoacetate (3h): Colorless liquid (88%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2960, 2839, 1730, 1671, 1595, 1512, 1250, 1155, 646. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.93 (d, J = 8.8 Hz, 2H), 7.38 (d, J = 8.8 Hz, 2H), 6.91 (t, J = 9.5 Hz, 4H), 5.33 (s, 2H), 3.86 (s, 3H), 3.8 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 184.6, 165.0, 164.1, 160.0, 132.6, 130.6, 126.8, 125.5, 114.3, 114.1, 67.5, 55.6, 55.3. HR-MS (ESI+) m/z calculated for $[C_{17}H_{16}NaO_5]^+$ = $[M+Na]^+$: 323.0890; found: 323.0889.



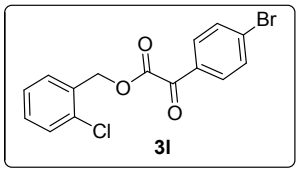
Butyl 2-(4-methoxy phenyl)-2-oxoacetate (3i):[5] Colorless liquid (90%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2961, 2874, 1731, 1673, 1595, 1263, 1158, 1016, 849, 769, 616. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.99 (d, J = 8.8 Hz, 2H), 6.98 (d, J = 8.8 Hz, 2H), 4.38 (t, J = 6.6 Hz, 2H), 3.89 (s, 3H), 1.76 (m, 2H), 1.45 (dq, J_a = 15.1 and J_b = 7.5 Hz, 2H), 0.97 (t, J = 7.3 Hz, 3H), ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 184.9, 165.0, 164.4, 125.5, 114.5, 65.9, 55.6, 30.5, 19.0, 13.6.



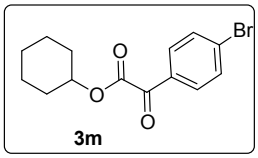
Isopropyl 2-(4-methoxy phenyl)-2-oxoacetate (3j):[4] Colorless liquid (90%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2983, 2938, 1726, 1672, 1595, 1263, 1207, 1161, 815, 645, 615. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.98 (m, 2H), 6.97 (m, 2H), 5.31 (dt, J_a = 12.7 and J_b = 6.4 Hz, 1H), 3.89 (s, 3H), 1.4 (d, J = 5.9 Hz, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 185.2, 164.9, 164.0, 132, 125.6, 114, 70.4, 55.6, 21.7.



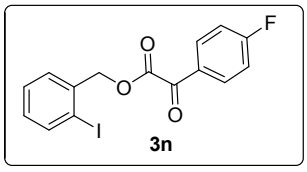
Benzyl 2-(4-methoxy phenyl)-2-oxoacetate (3k):[6] Colorless liquid (95%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2937, 2842, 1731, 1671, 1594, 1262, 1199, 1156, 697, 647. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.95 (m, 2H), 7.43 (m, 2H), 7.38 (m, 3H), 6.94 (d, J = 9.3 Hz, 2H), 5.39 (s, 2H), 3.87 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 184.5, 165.0, 164.0, 134.7, 132.6, 128.8, 128.6, 125.5, 114.3, 67.6, 55.7.



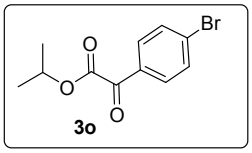
2-Chlorobenzyl 2-(4-bromophenyl)-2-oxoacetate (3l): Colorless liquid (89%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2957, 2923, 1737, 1688, 1585, 1193, 1171, 996, 846, 754. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.89 (d, J = 8.3 Hz, 2H), 7.66 (d, J = 8.8 Hz, 2H), 7.5 (m, 1H), 7.43 (m, 1H), 7.32 (m, 2H), 5.52 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 184.7, 162.8, 134.0, 132, 132, 131.5, 130.7, 130.4, 130.3, 129.8, 127.1, 65.2. HR-MS (ESI+) m/z calculated for $[C_{15}H_{10}BrClNaO_3]^+$ = $[M+Na]^+$: 376.9373; found: 376.9371.



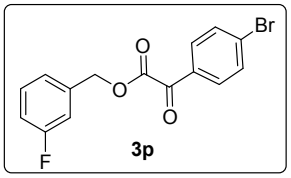
Cyclohexyl 2-(4-bromophenyl)-2-oxoacetate (3m): Colorless liquid (85%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2936, 2859, 1724, 1686, 1303, 1198, 1172, 1069, 987, 851, 768. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.8 (d, J = 8.3 Hz, 2H), 7.58 (d, J = 8.8 Hz, 2H), 5.01 (m, 1H), 1.92 (m, 2H), 1.72 (m, 2H), 1.53 (m, 3H), 1.35 (dtt, J_a = 13.6, J_b = 10.2 and J_c = 3.3 Hz, 2H), 1.24 (m, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 186.5, 163.0, 132.3, 131.4, 131.4, 131.4, 31.4, 25.1, 23.6. HR-MS (ESI+) m/z calculated for $[C_{14}H_{19}NBrO_3]^+$ = $[M+NH_4]^+$: 328.0543; found: 328.0541.



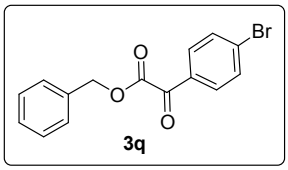
2-Iodobenzyl 2-(4-fluorophenyl)-2-oxoacetate (3n): Colorless liquid (90%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 3070, 2924, 1737, 1687, 1597, 1187, 996, 853, 750, 615. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 8.1 (m, 2H), 7.89 (d, J = 7.3Hz, 1H), 7.48 (dd, J_a = 7.8 and J_b = 1.5 Hz, 1H), 7.39 (m, 1H), 7.19 (m, 2H), 7.07 (td, J_a = 7.7 and J_b = 1.7 Hz, 1H), 5.44 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 184.0, 163.0, 139.7, 136.9, 133.1, 133.0, 130.5, 130.0, 128.9, 128.6, 116.5, 116.2, 98.6, 71.6. HR-MS (ESI+) m/z calculated for $[C_{15}H_{14}INFO_3]^+ = [M+NH_4]^+$: 401.9997; found: 401.9995.



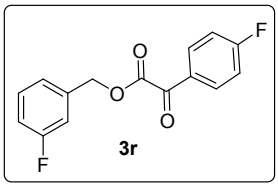
Isopropyl 2-(4-bromophenyl)-2-oxoacetate (3o):[4] Light yellow oil (89%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2983, 2936, 1727, 1683, 1582, 1309, 1198, 1069, 985, 805, 764. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.88 (d, J = 8.3 Hz, 2H), 7.66 (d, J = 8.3 Hz, 2H), 5.32 (sept, J = 6.3 Hz, 1H), 1.41 (d, J = 6.4 Hz, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 185.4, 163.0, 132.3, 131.4, 130.4, 71.0, 21.7.



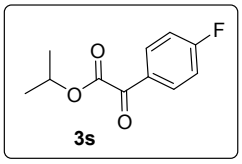
3-Fluorobenzyl 2-(4-bromphenyl)-2-oxoacetate (3p): Colorless liquid (90%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2926, 1734, 1687, 1585, 1259, 1170, 1070, 981, 782, 683. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.87 (d, J = 8.3 Hz, 2H), 7.65 (d, J = 8.8 Hz, 2H), 7.37 (td, J_a = 7.8 and J_b = 5.9 Hz, 1H), 7.21 (d, J = 7.8 Hz, 1H), 7.15 (d, J = 9.3 Hz, 1H), 7.07 (td, J_a = 8.4 and J_b = 2.2 Hz, 1H), 5.39 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 184.5, 164.1, 162.7, 161.6, 136.8, 136.7, 132.4, 131.4, 131.2, 130.8, 130.4, 124.0, 116.0, 115.4, 67.0. HR-MS (ESI+) m/z calculated for $[C_{15}H_{10}BrNaFO_3]^+ = [M+Na]^+$: 358.9690; found: 358.9688.



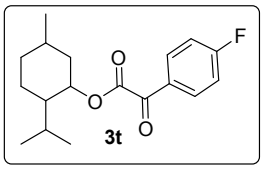
Benzyl 2-(4-bromphenyl)-2-oxoacetate (3q):[7] Colorless liquid (93%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2924, 2852, 1734, 1685, 1595, 1237, 1183, 749, 648, 616. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 7.85 (d, J = 8.3 Hz, 2H), 7.63 (d, J = 8.3 Hz, 2H), 7.44 (m, 2H), 7.4 (m, 3H), 5.41 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 184.5, 163.0, 134.4, 132.3, 131.4, 131.3, 130.6, 129.7, 128.9, 128.8, 128.7, 68.0.



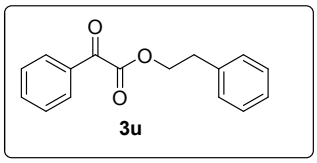
3-Fluorobenzyl 2-(4-fluorophenyl)-2-oxoacetate (3r): Colorless liquid (91%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2925, 1734, 1686, 1594, 1238, 1185, 983, 784, 614, 542. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 8.05 (m, 2H), 7.37 (td, J_a = 7.8 and J_b = 5.9 Hz, 1H), 7.18 (m, 4H), 7.06 (td, J_a = 8.4 and J_b = 2.2 Hz, 1H), 5.39 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 183.9, 168.2, 165.6, 164.1, 163.0, 161.6, 136.8, 136.8, 133.0, 132.9, 130.5, 130.4, 128.9, 124.0, 116.5, 116.2, 115.9, 115.7, 115.5, 115.3, 66.9. HR-MS (ESI+) m/z calculated for $[C_{15}H_{10}F_2NaO_3]^+$ = $[M+Na]^+$: 299.0489; found: 299.0490.



Isopropyl 2-(4-fluorophenyl)-2-oxoacetate (3s):[4] Light yellow liquid (90%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2985, 2935, 1728, 1686, 1596, 1296, 1232, 1198, 988, 858, 676, 613. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 8.06 (m, 2H), 7.19 (t, J = 8.6 Hz, 2H), 5.32 (sept, 1H), 1.42 (d, J = 6.4 Hz, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 184.9, 168.0, 165.5, 163.2, 132.9, 132.8, 129.1, 116.4, 116.1, 70.9, 21.7.

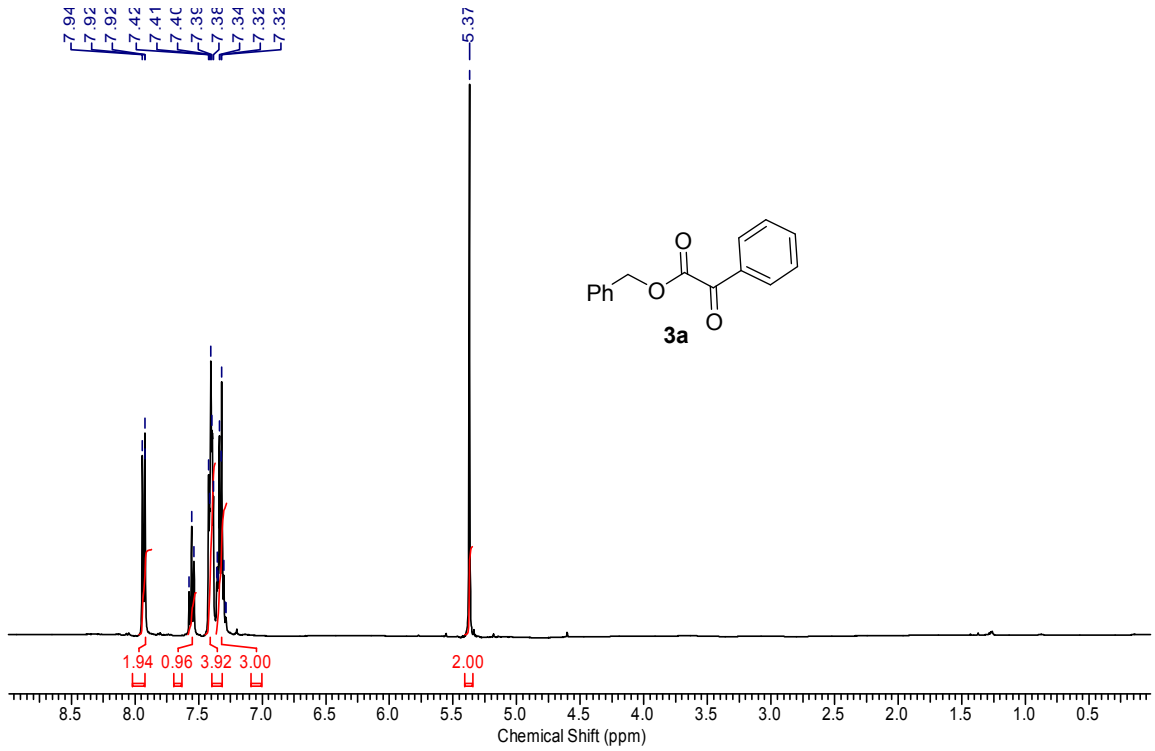


2-Hydroxy-3,3,6-trimethylcyclohexyl 2-(4-fluorophenyl)-2-oxoacetate (3t):[8] Colorless liquid (85%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 2956, 2871, 1724, 1688, 1597, 1238, 1198, 1153, 734, 636, 617. ^1H NMR ($CDCl_3$, 400 MHz): δ_H = 8.04 (m, 2H), 7.2 (m, 2H), 5.0 (td, J_a = 10.9 and J_b = 4.6 Hz, 2H), 2.16 (m, 1H), 1.94 (dtd, J_a = 13.9, J_b = 7 and J_c = 2.9 Hz, 1H), 1.75 (m, 2H), 1.55 (m, 2H), 1.15 (m, 2H), 0.96 (d, J = 6.4 Hz, 3H), 0.9 (d, J = 6.8 Hz, 3H), 0.84 (d, J = 6.8 Hz, 2H).^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 185.0, 168.0,165.5, 163.5, 132.9, 132.7, 129.1, 116.4, 116.2, 77.1, 46.8, 40.6, 34.0, 31.6, 26.2, 23.3, 22.0, 20.7, 16.2.

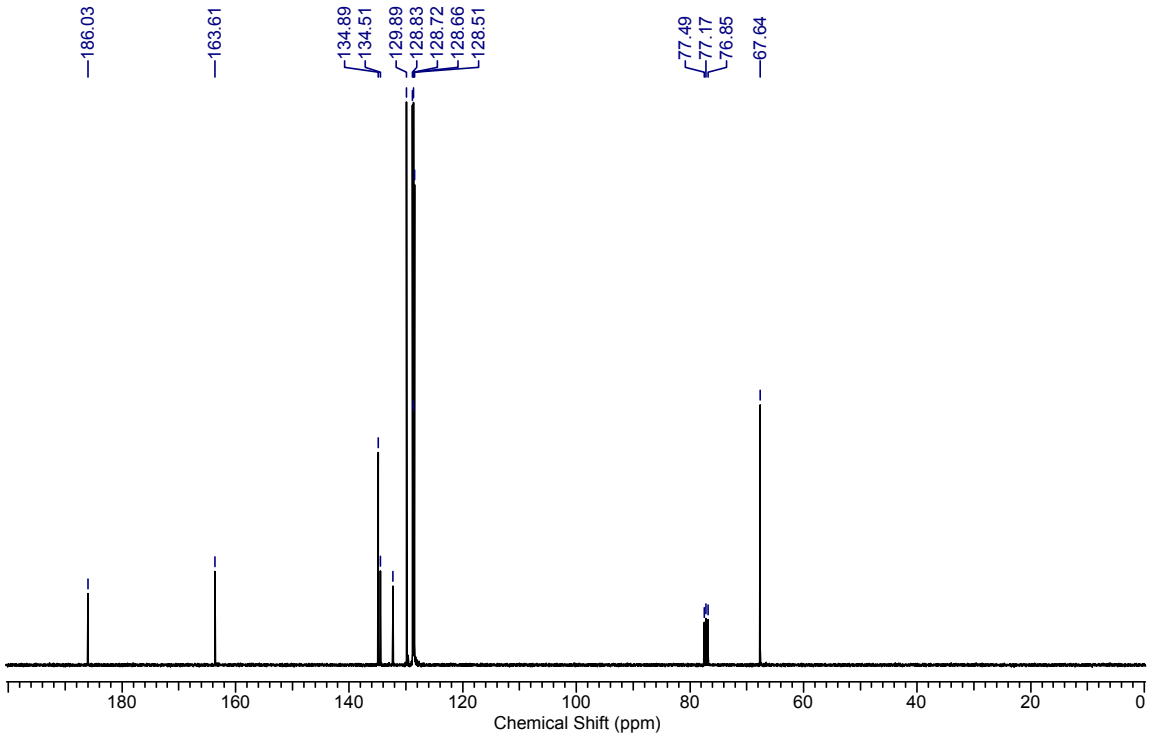


Phenethyl 2-oxo-2-phenylacetate (3u):[2] Colorless liquid (95%), IR (MIR-ATR, 4000–600 cm^{-1}): υ_{max} = 3028, 1735, 1687, 1322, 1173, 992, 744, 666, 570. ^{1}H NMR ($CDCl_3$, 400 MHz): δ_H = 7.84 (d, J = 7.8 Hz, 2H), 7.61 (m, 1H), 7.43 (m, 2H), 7.28 (m, 5H), 4.61 (t, J = 6.8 Hz, 2H), 3.07 (t, J = 6.8 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ_C = 186.4, 163.8, 137.0, 134.9, 132.3, 130.0, 129.0, 128.9, 128.7, 126.9, 66.4, 35.0.

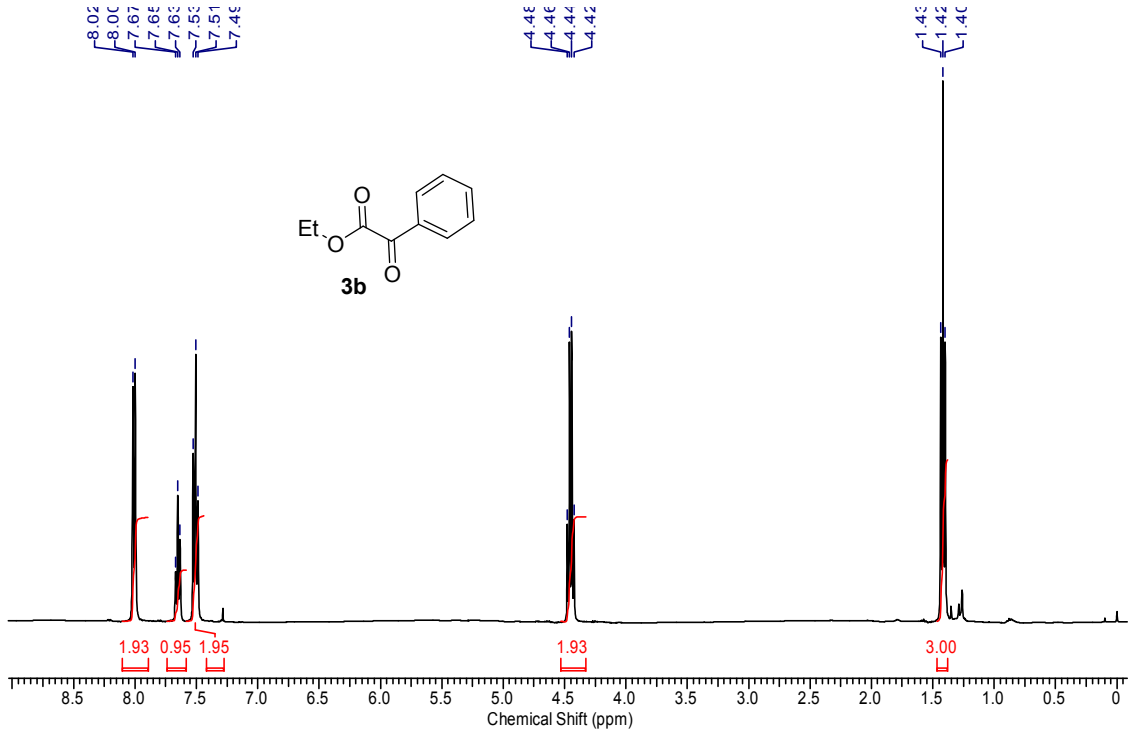
Copies of ^{1}H NMR & ^{13}C NMR Spectra of all Compounds (**3a-3u**)



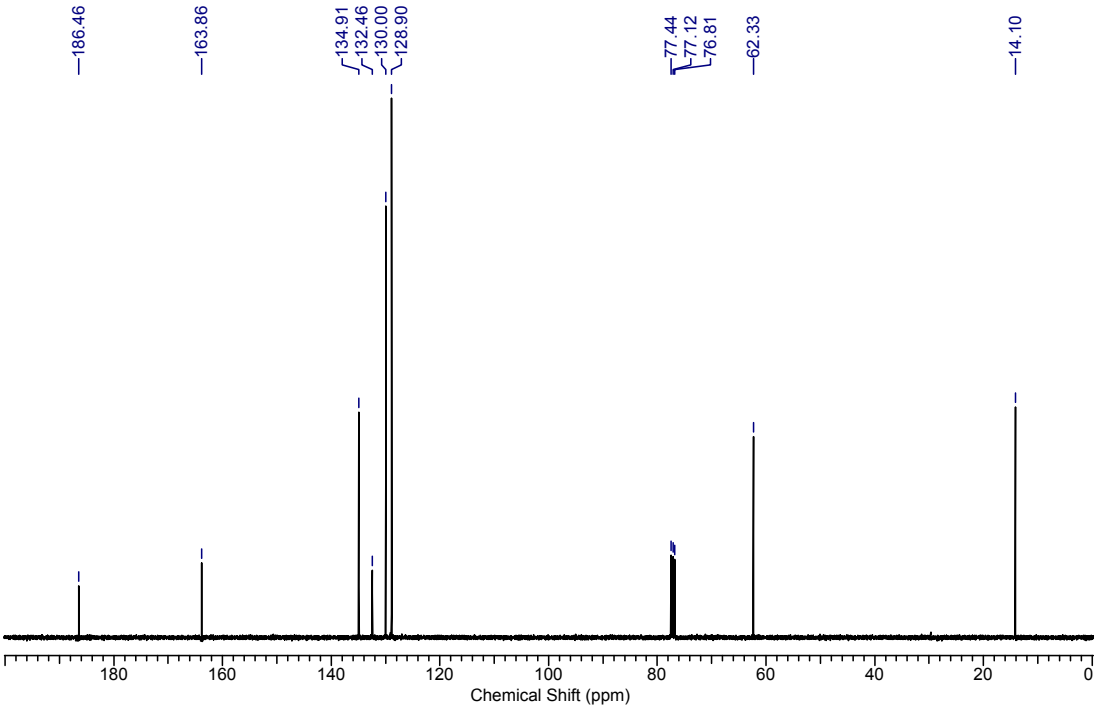
^{1}H NMR (400 MHz) spectrum of compound **3a** in $CDCl_3$



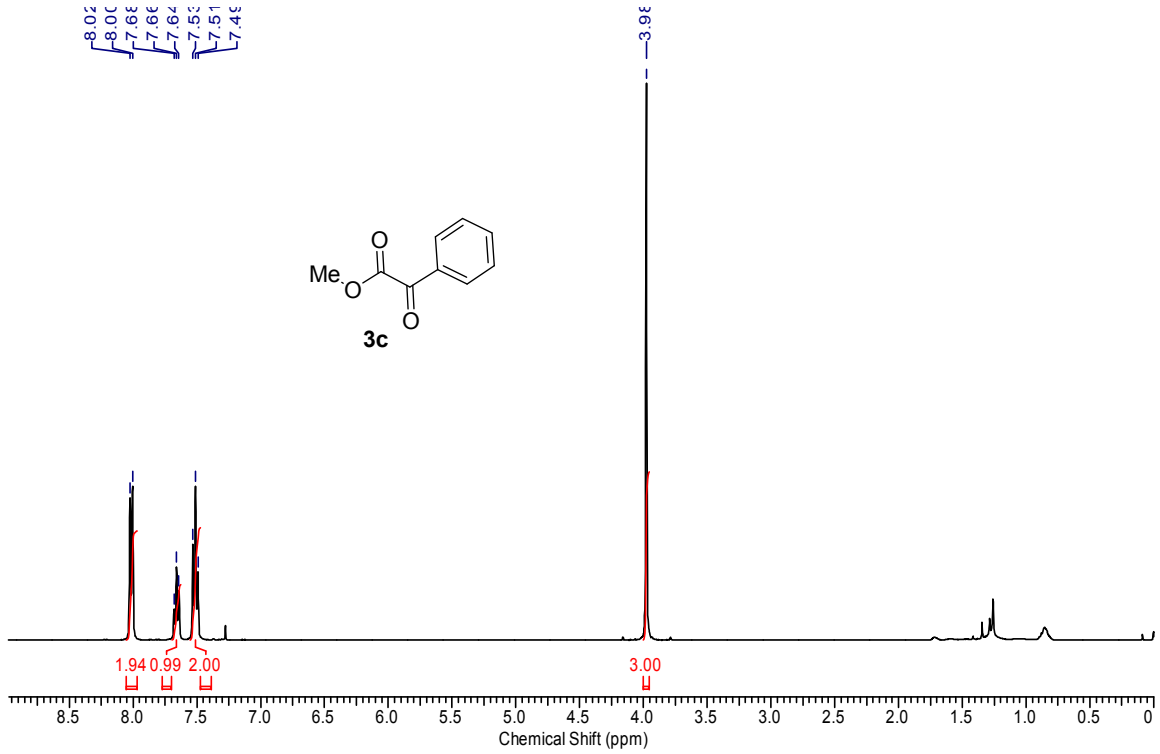
^{13}C NMR (100 MHz) spectrum of compound **3a** in $CDCl_3$



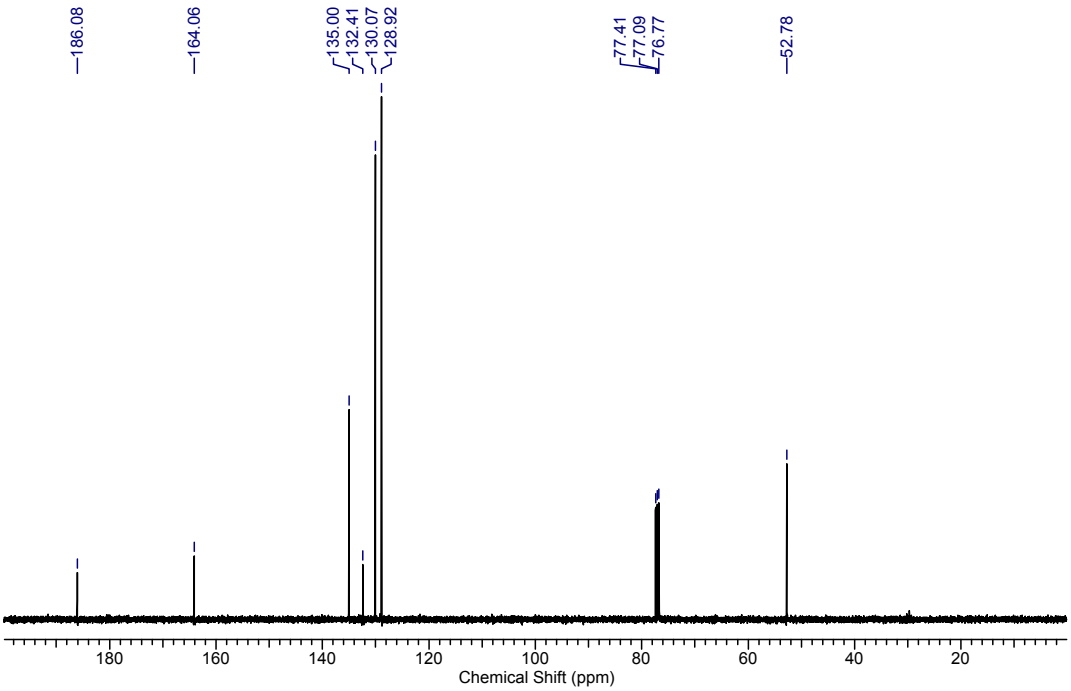
^{1}H NMR (400 MHz) spectrum of compound **3b** in $CDCl_3$



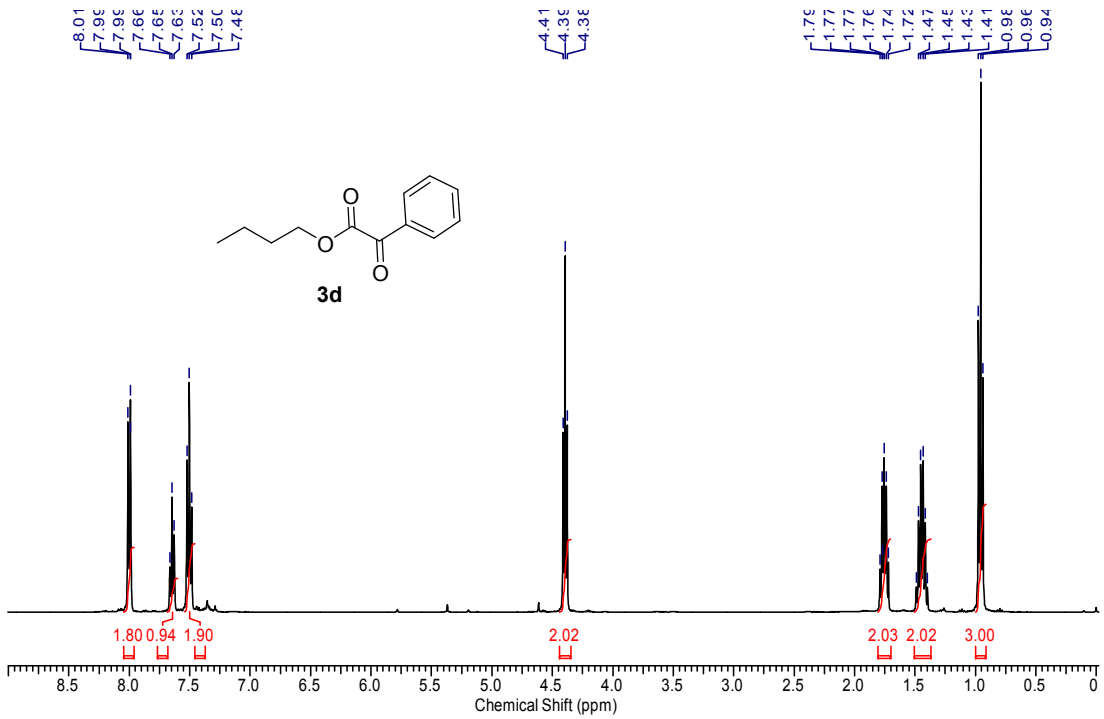
^{13}C NMR (100 MHz) spectrum of compound **3b** in $CDCl_3$



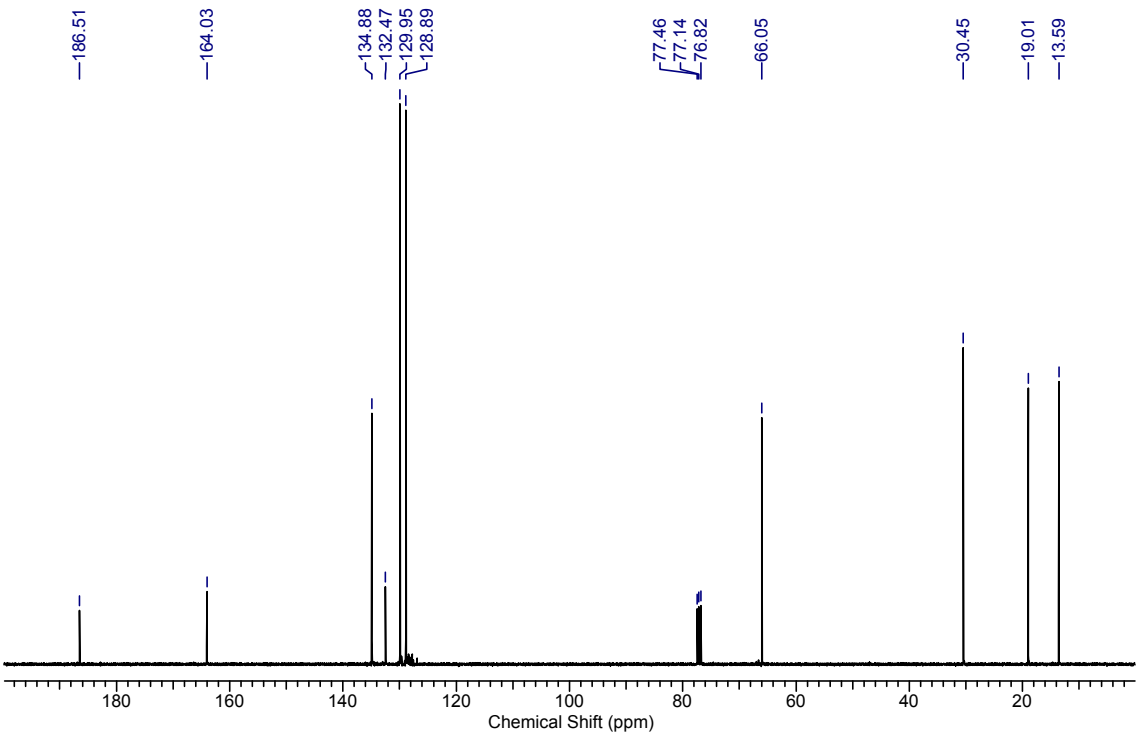
^{1}H NMR (400 MHz) spectrum of compound **3c** in $CDCl_3$



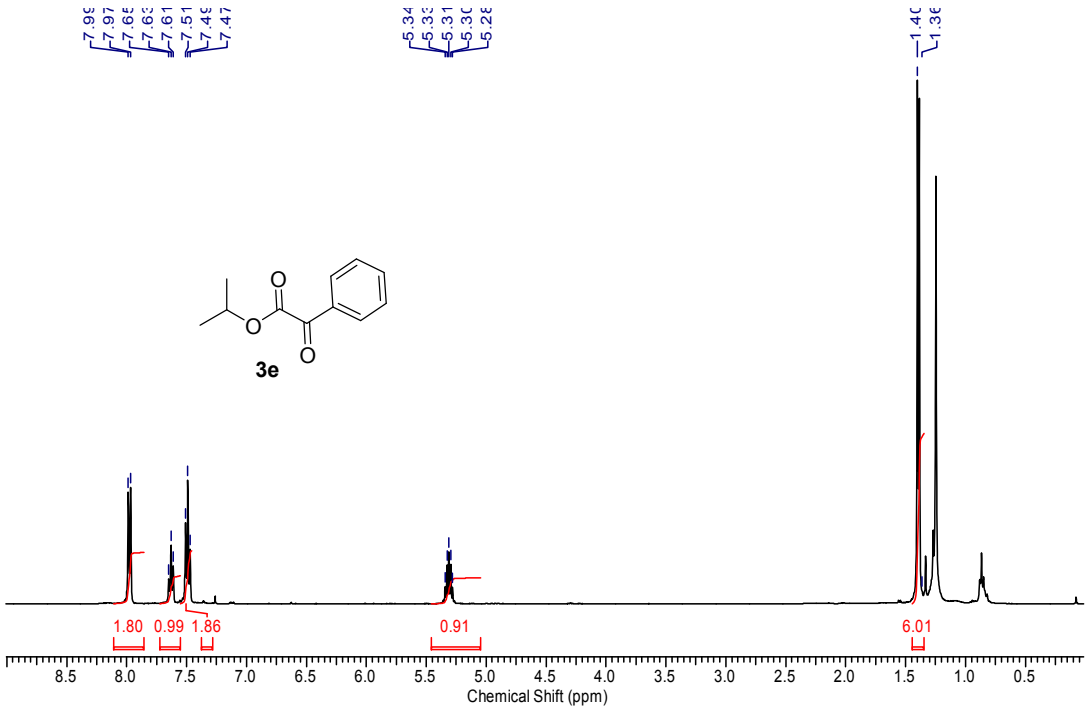
^{13}C NMR (100 MHz) spectrum of compound **3c** in $CDCl_3$



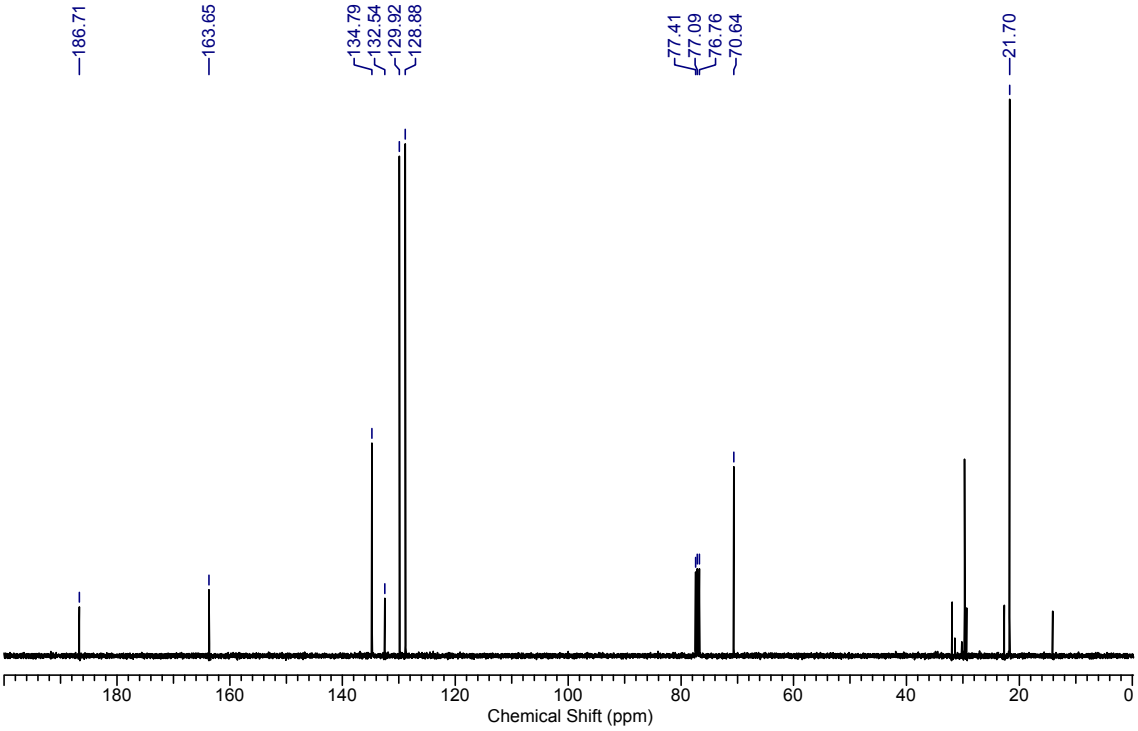
^{1}H NMR (400 MHz) spectrum of compound **3d** in $CDCl_3$



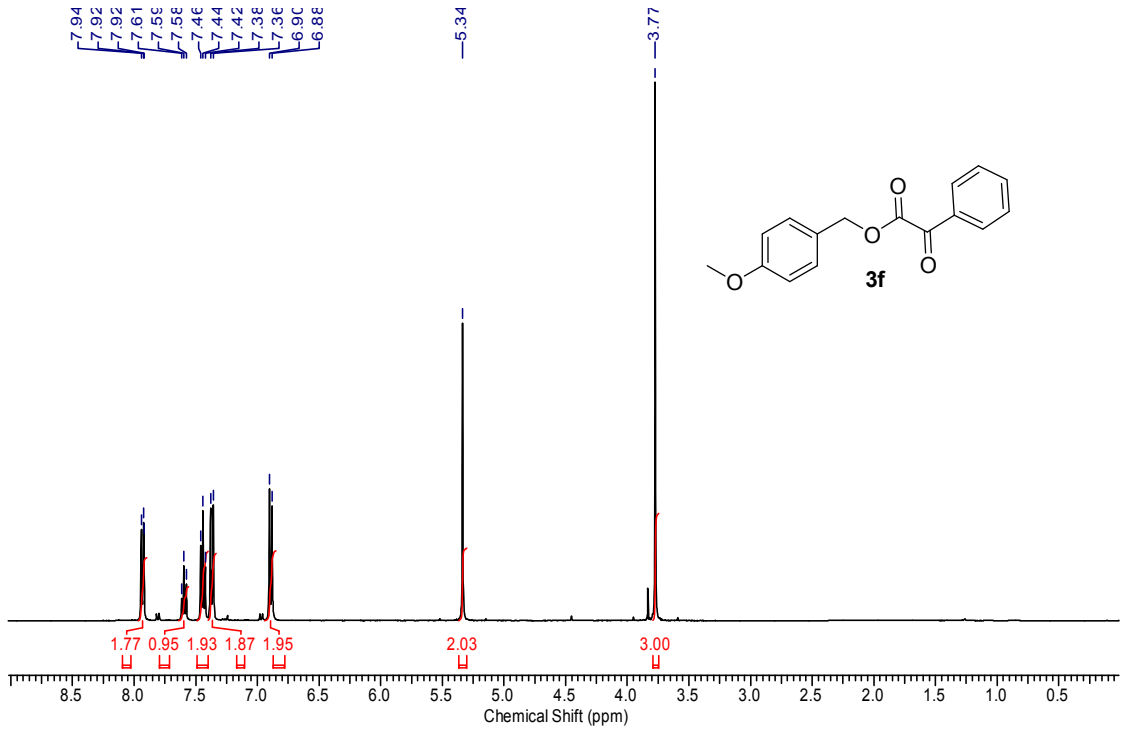
^{13}C NMR (100 MHz) spectrum of compound **3d** in $CDCl_3$



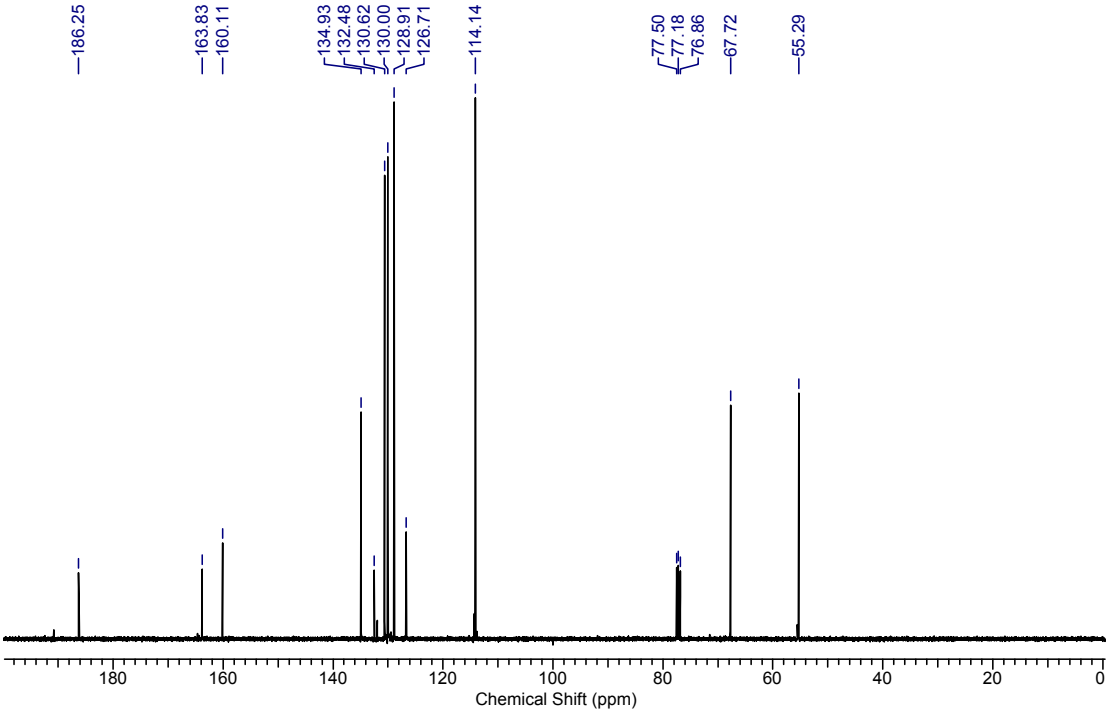
^{1}H NMR (400 MHz) spectrum of compound **3e** in $CDCl_3$



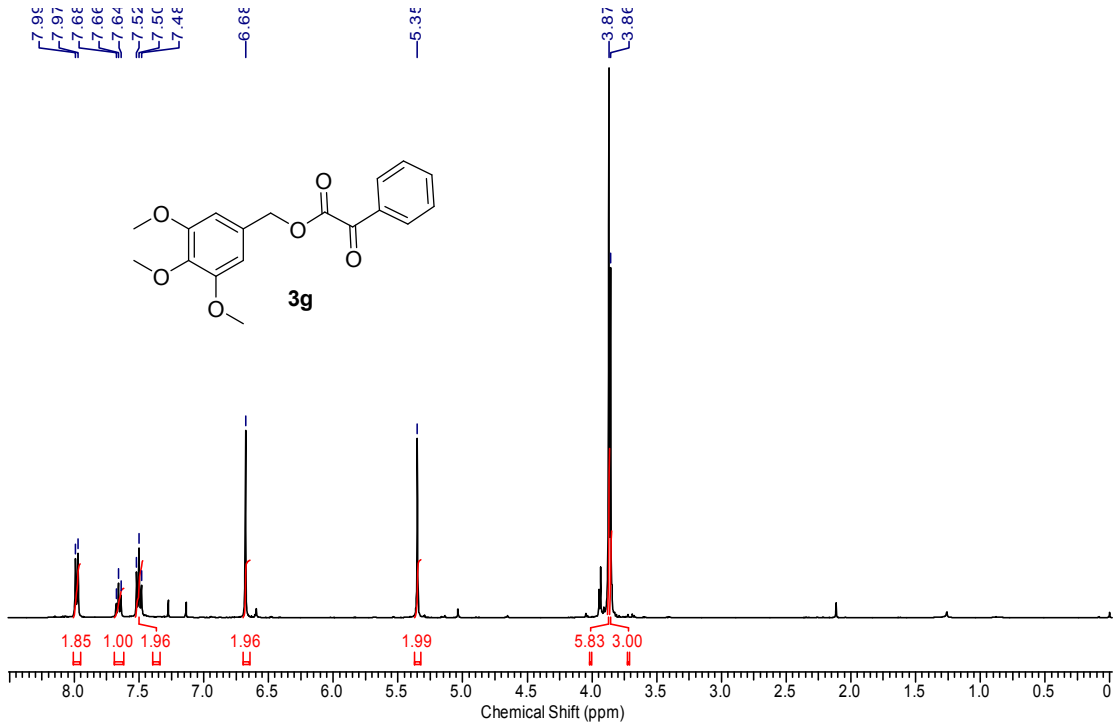
^{13}C NMR (100 MHz) spectrum of compound **3e** in $CDCl_3$



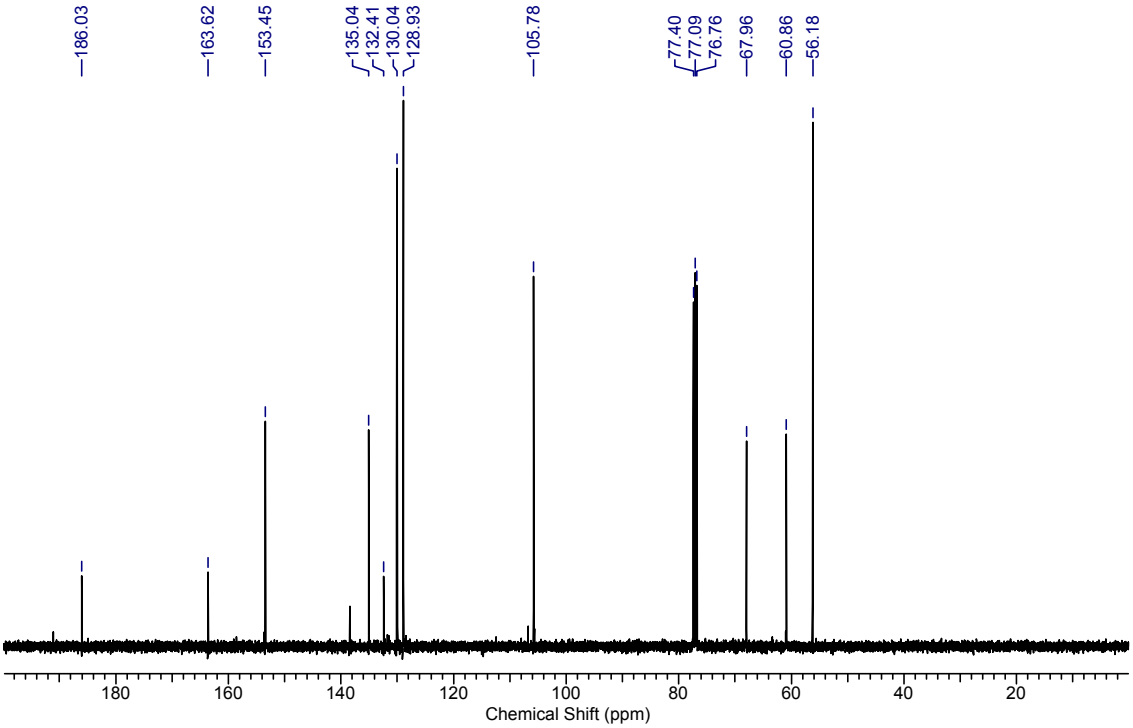
^{1}H NMR (400 MHz) spectrum of compound **3f** in $CDCl_3$



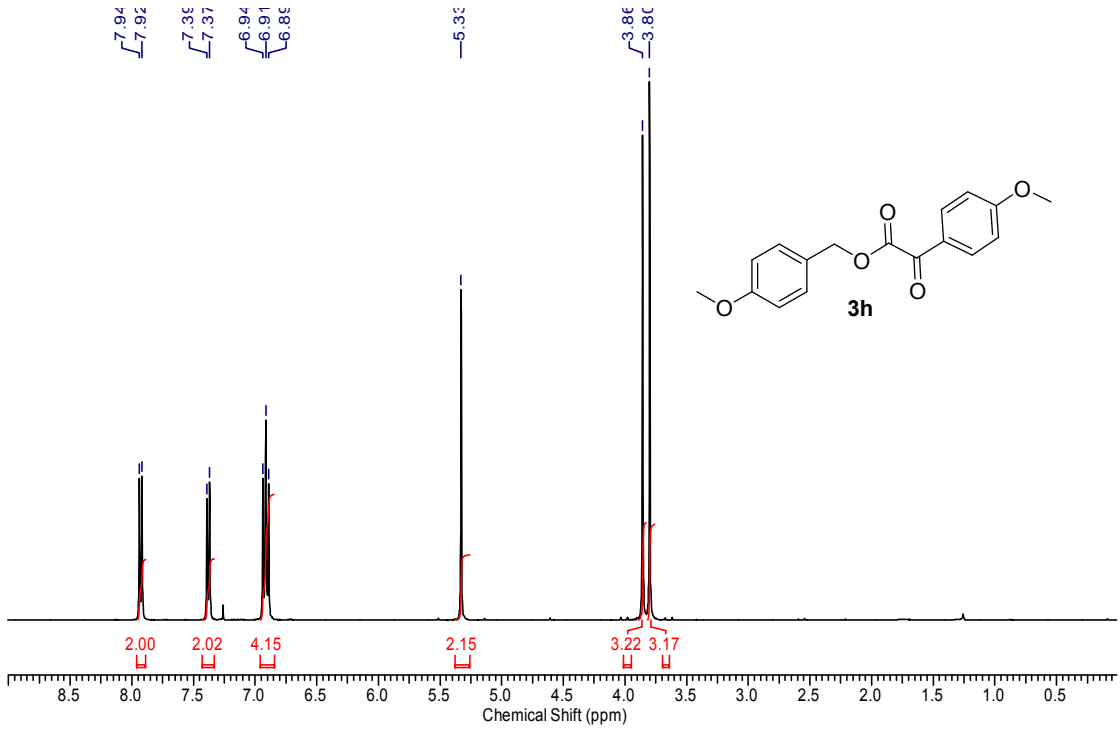
^{13}C NMR (100 MHz) spectrum of compound **3f** in $CDCl_3$



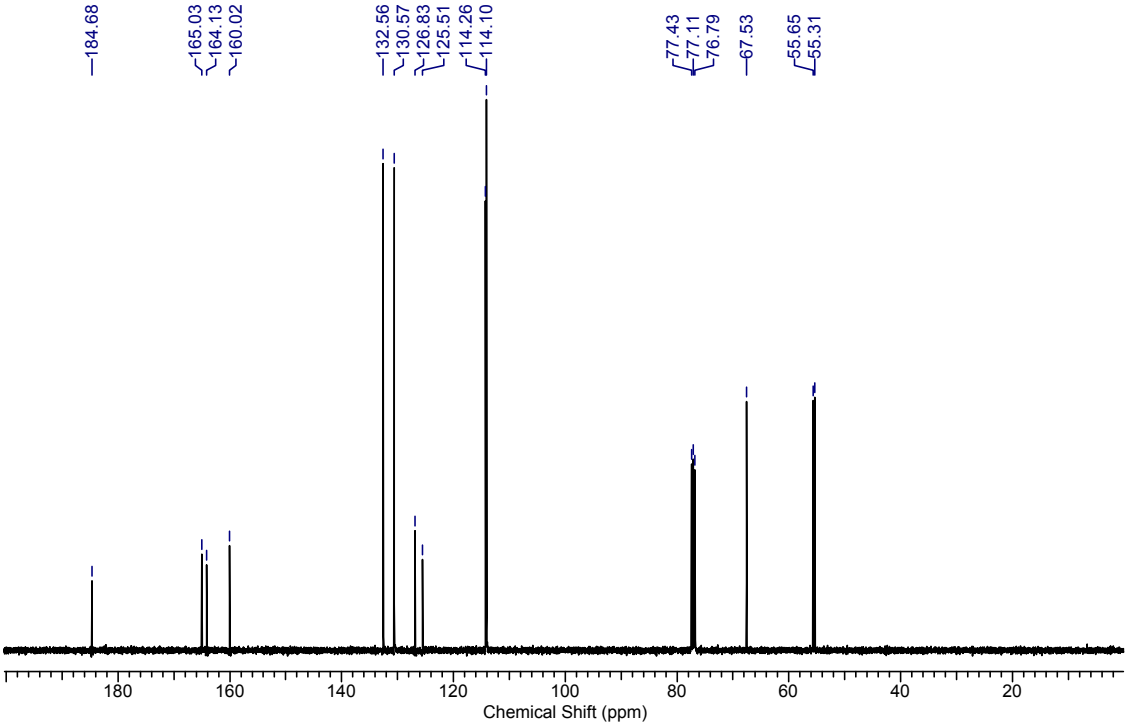
^{1}H NMR (400 MHz) spectrum of compound **3g** in $CDCl_3$



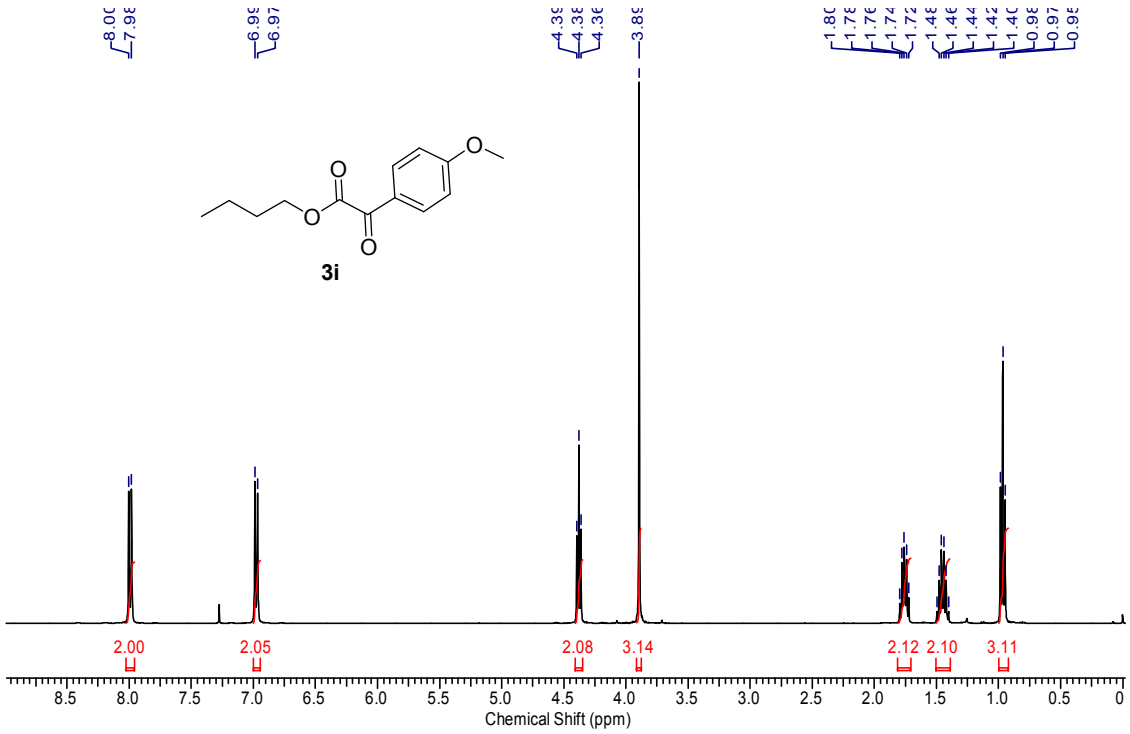
^{13}C NMR (100 MHz) spectrum of compound **3g** in $CDCl_3$



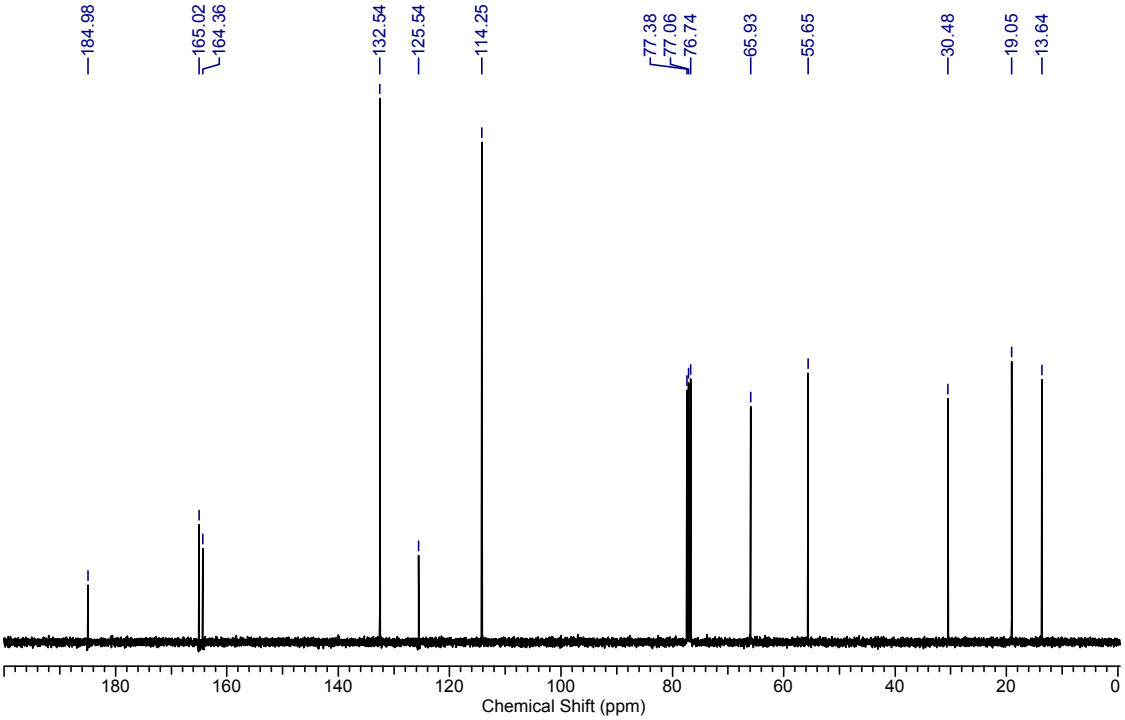
^{1}H NMR (400 MHz) spectrum of compound **3h** in $CDCl_3$



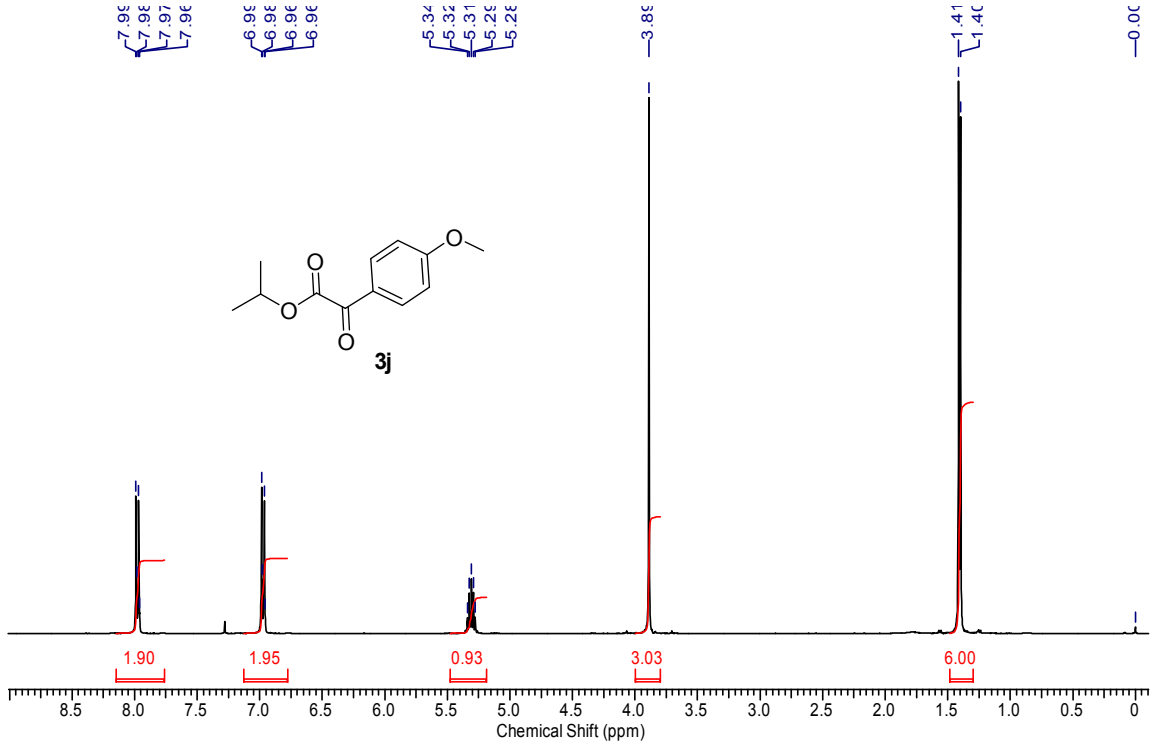
^{13}C NMR (100 MHz) spectrum of compound **3h** in $CDCl_3$



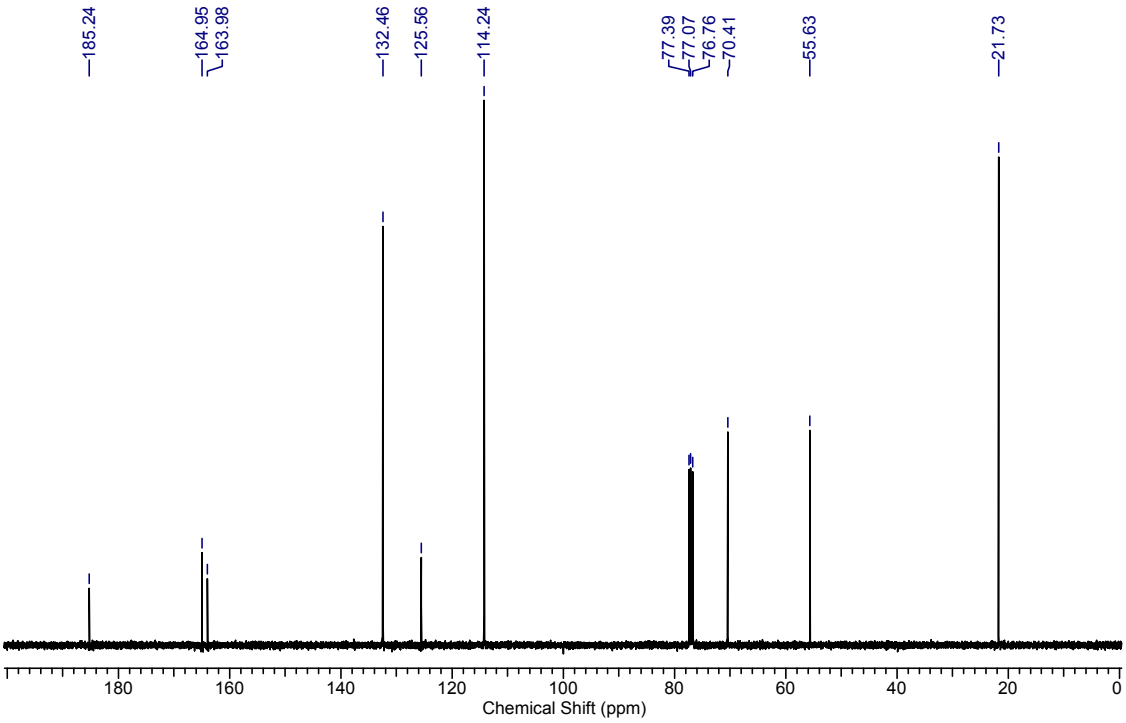
^{1}H NMR (400 MHz) spectrum of compound **3i** in $CDCl_3$



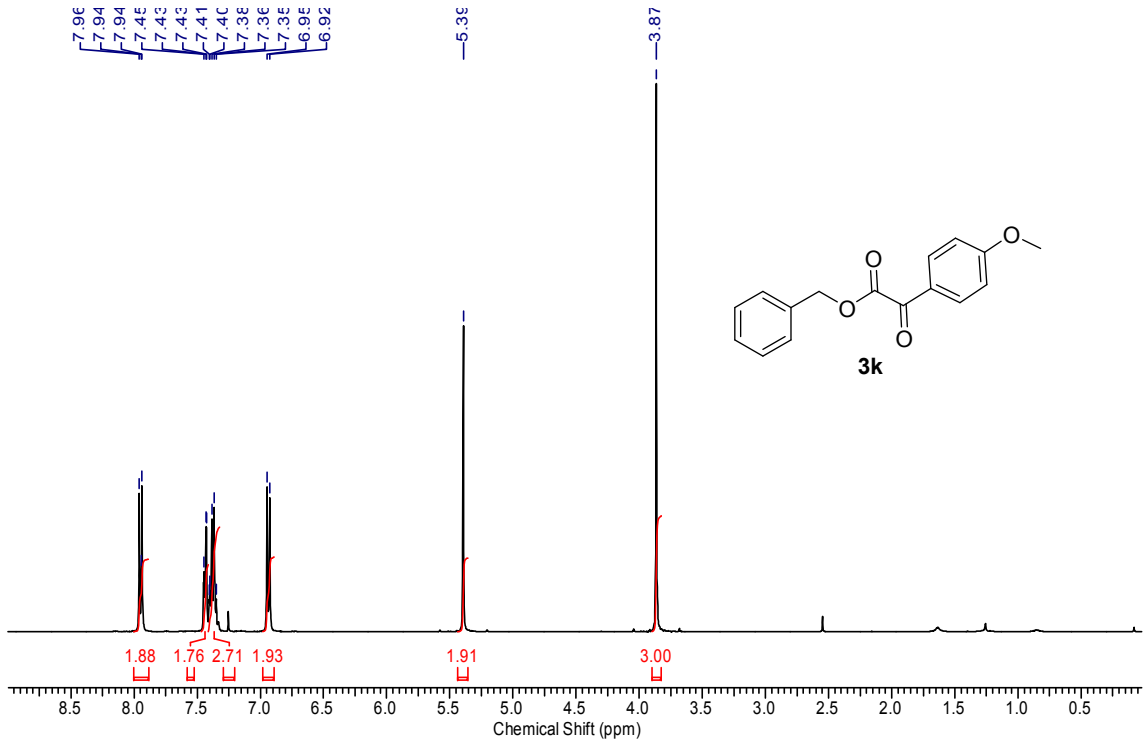
^{13}C NMR (100 MHz) spectrum of compound **3i** in $CDCl_3$



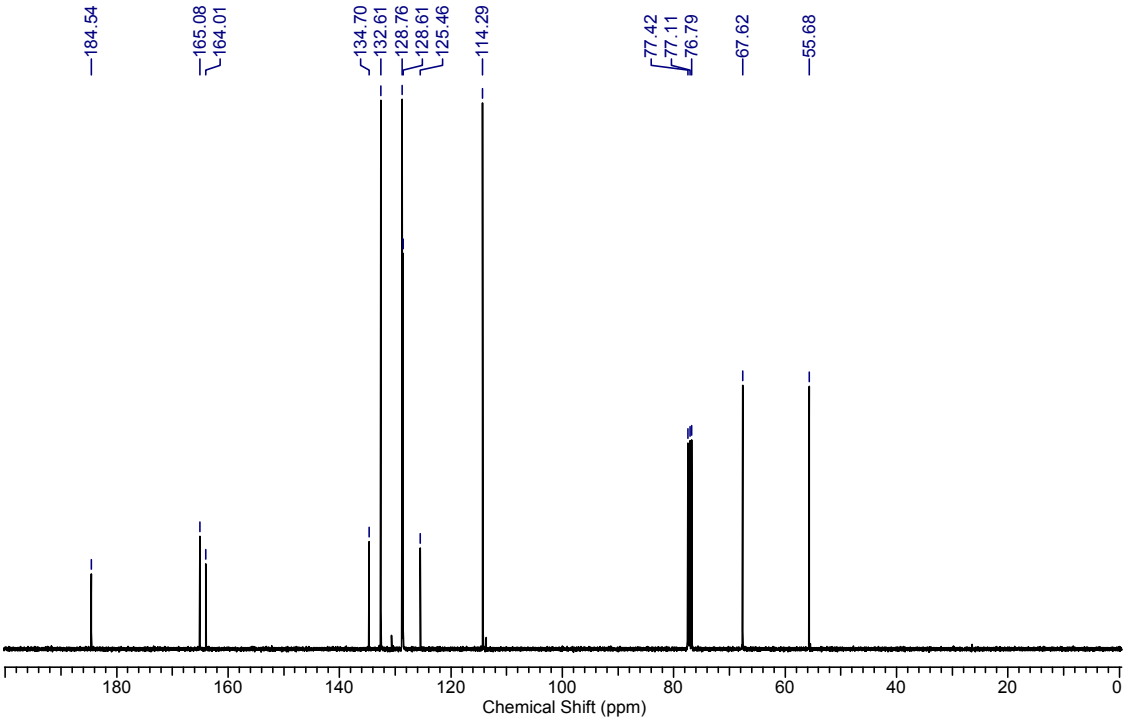
^{1}H NMR (400 MHz) spectrum of compound **3j** in $CDCl_3$



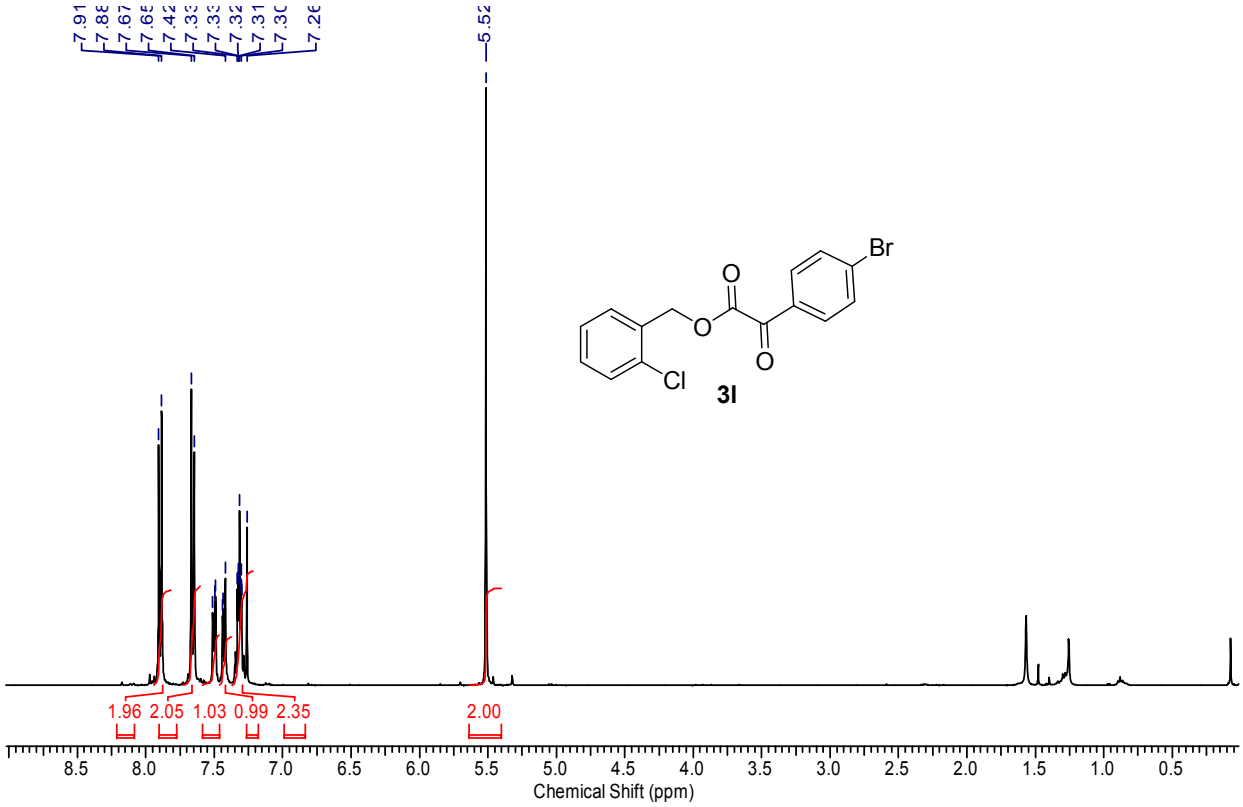
^{13}C NMR (100 MHz) spectrum of compound **3j** in $CDCl_3$



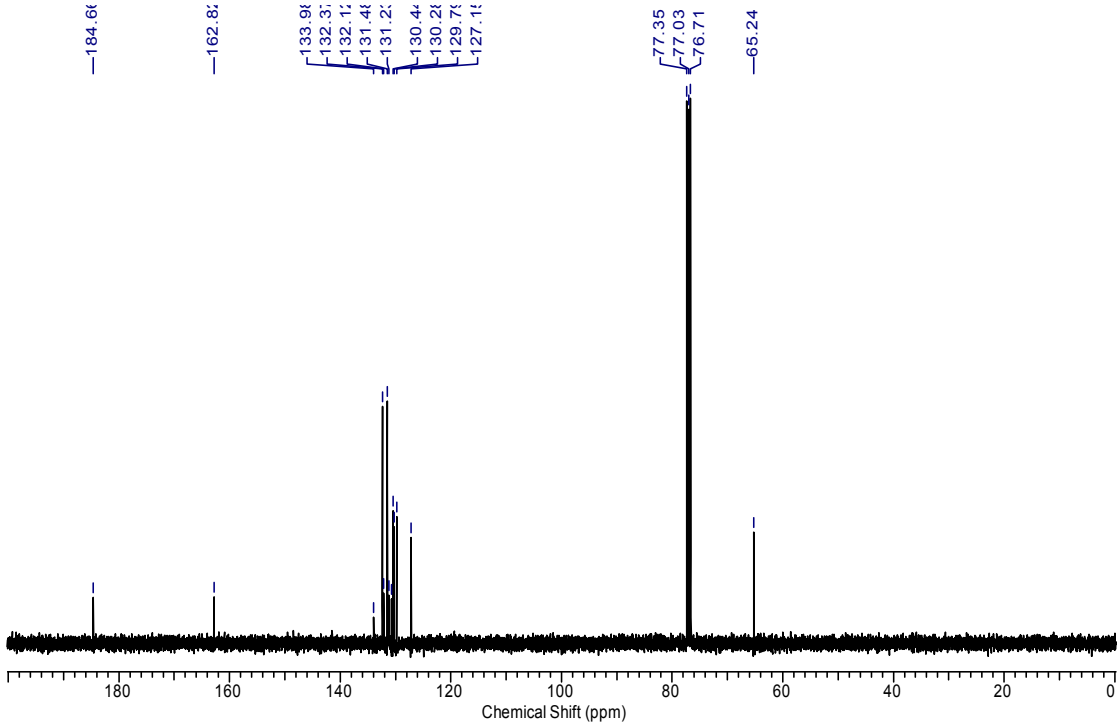
^{1}H NMR (400 MHz) spectrum of compound **3k** in $CDCl_3$



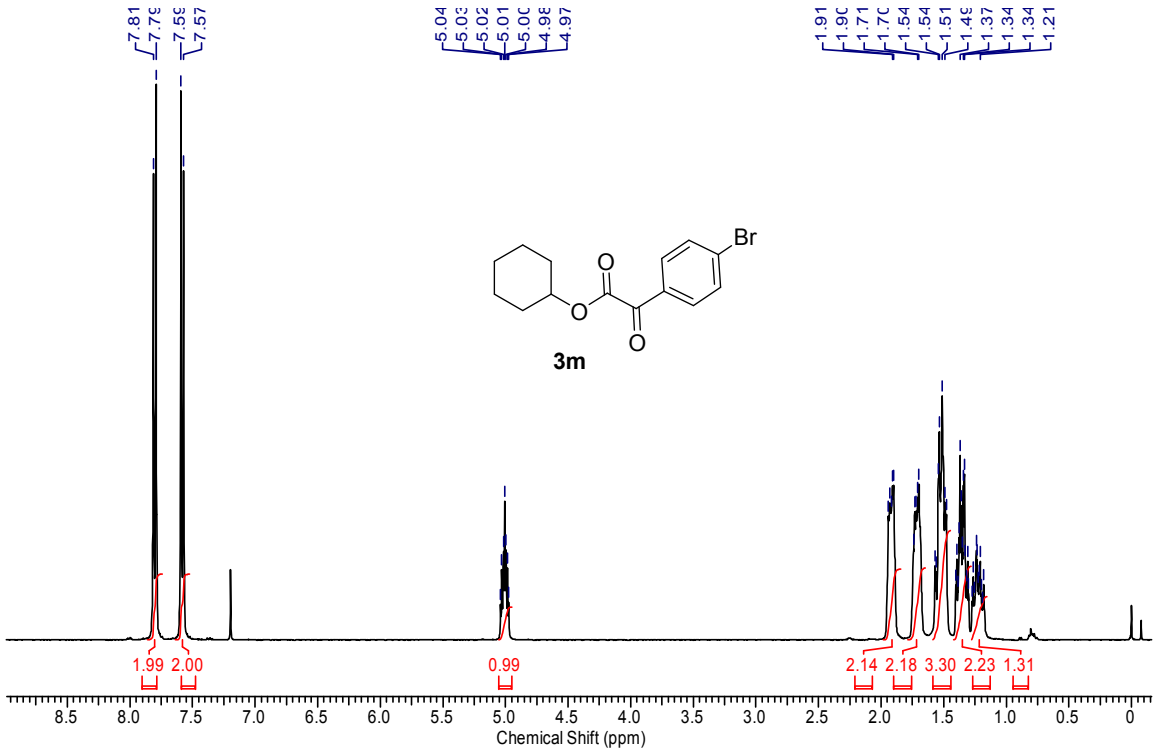
^{13}C NMR (100 MHz) spectrum of compound **3k** in $CDCl_3$



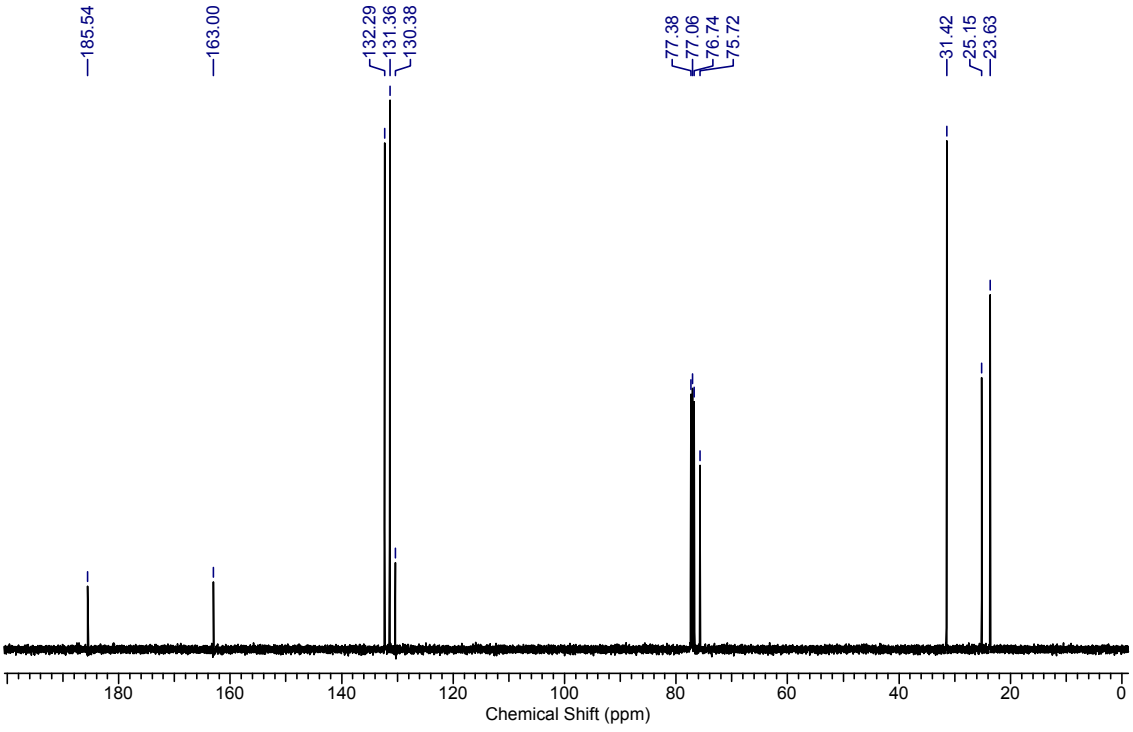
^{1}H NMR (400 MHz) spectrum of compound **3l** in $CDCl_3$



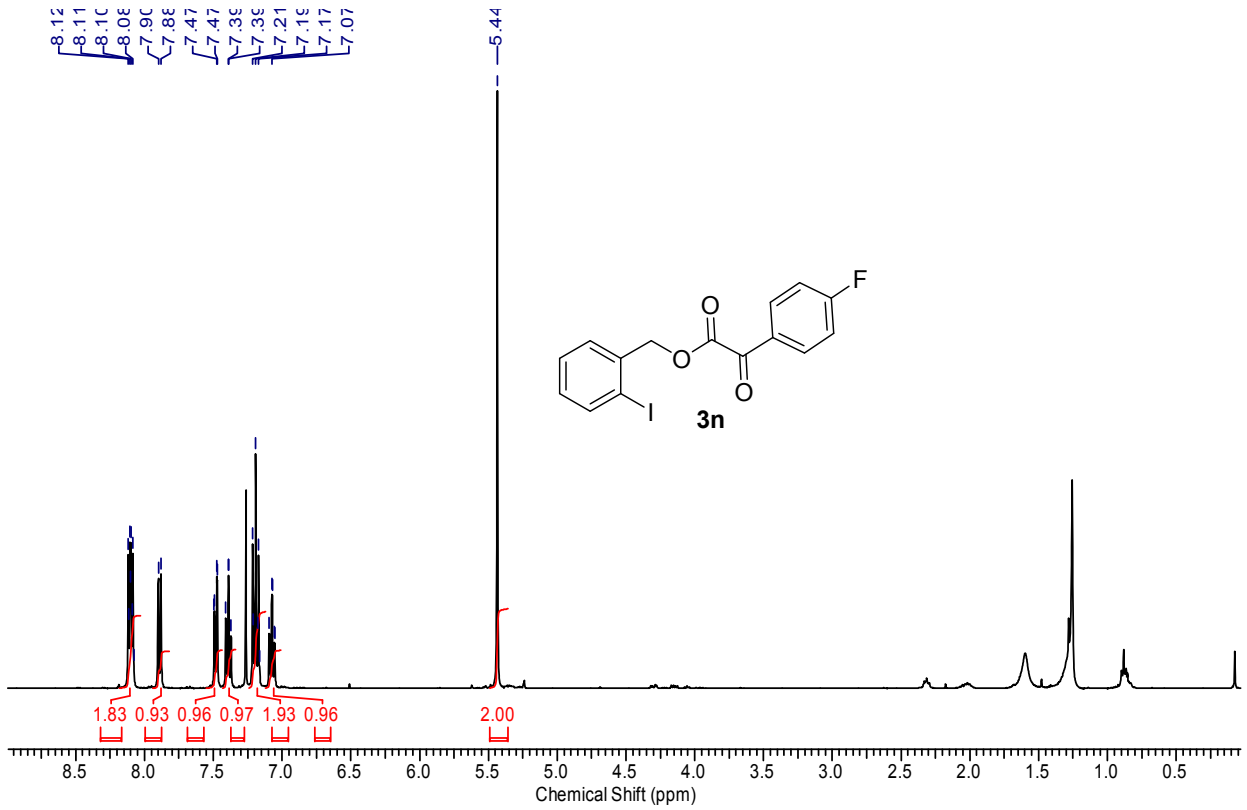
^{13}C NMR (100 MHz) spectrum of compound **3l** in $CDCl_3$



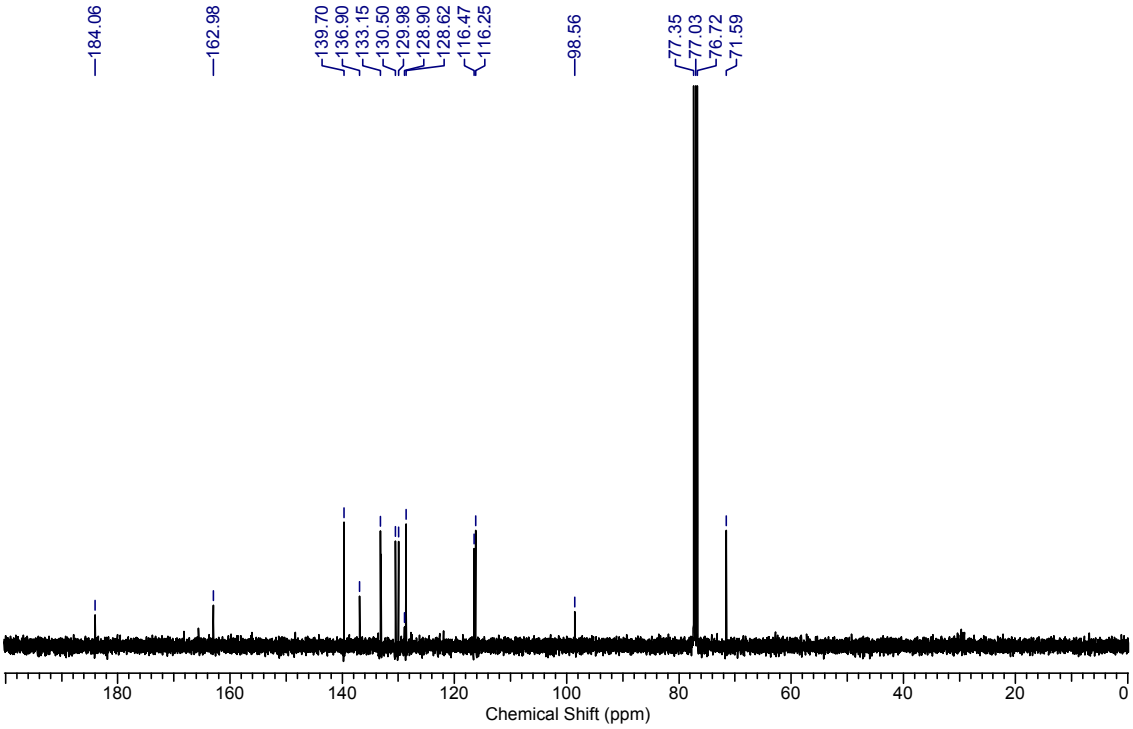
^{1}H NMR (400 MHz) spectrum of compound **3m** in $CDCl_3$



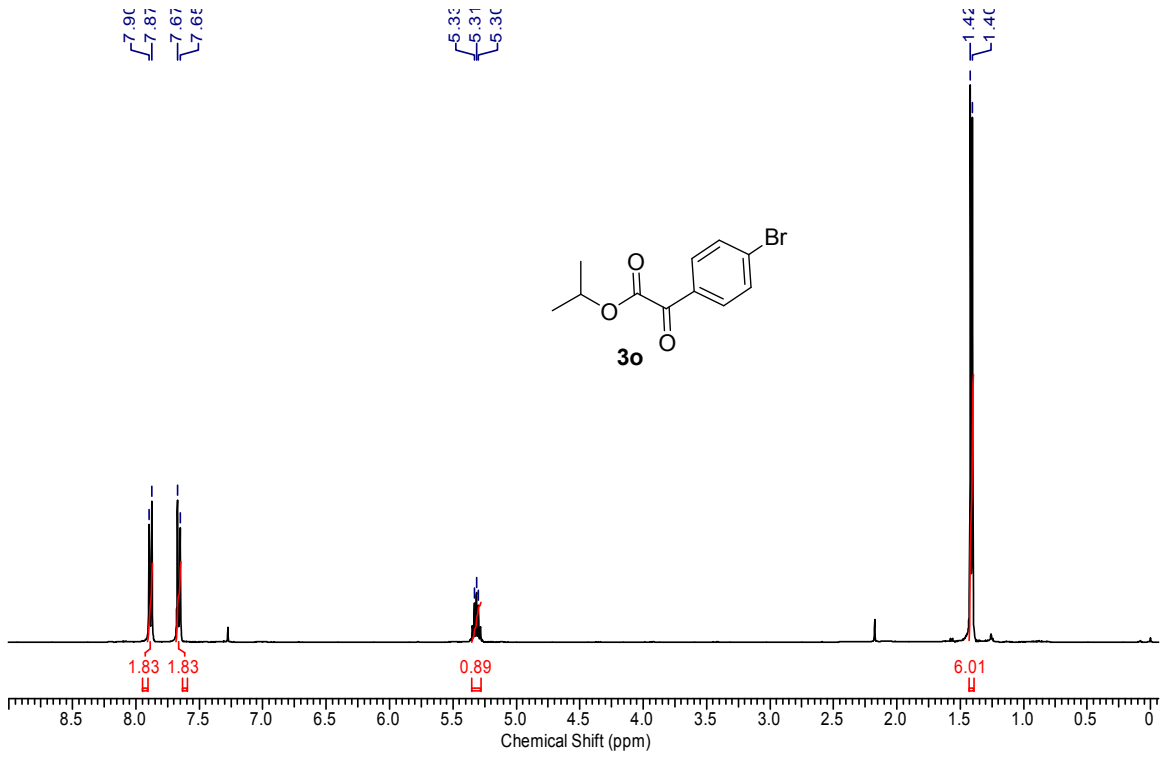
^{13}C NMR (100 MHz) spectrum of compound **3m** in $CDCl_3$



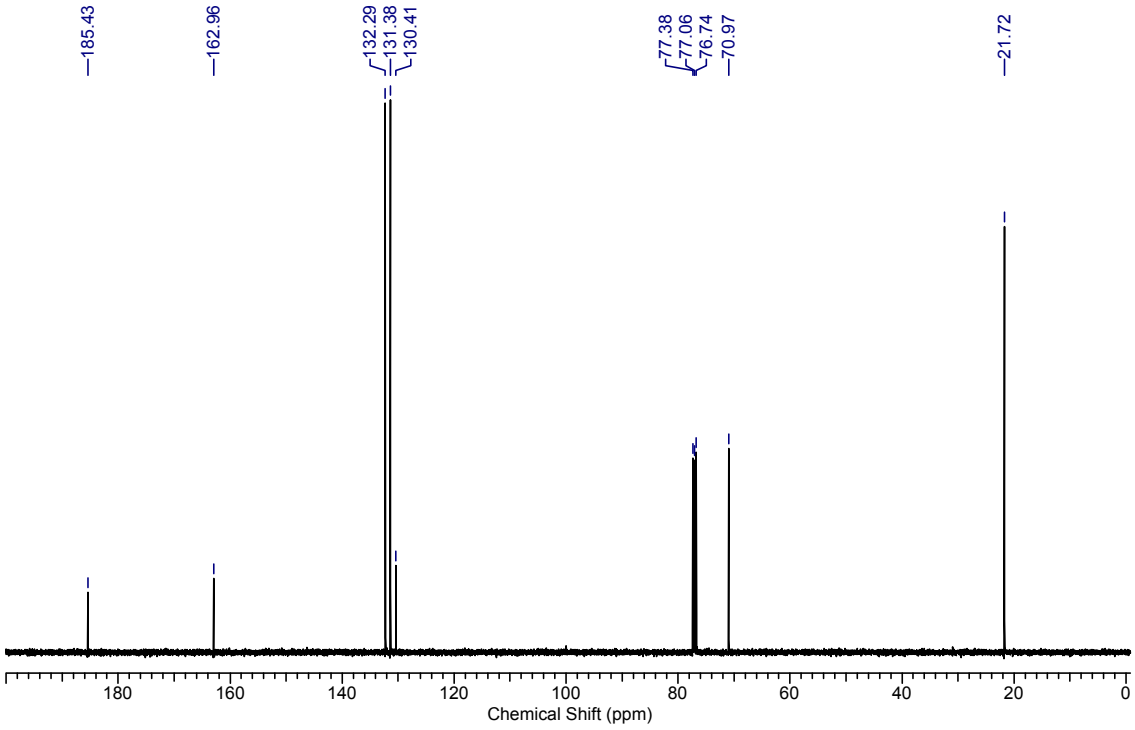
^{1}H NMR (400 MHz) spectrum of compound **3n** in $CDCl_3$



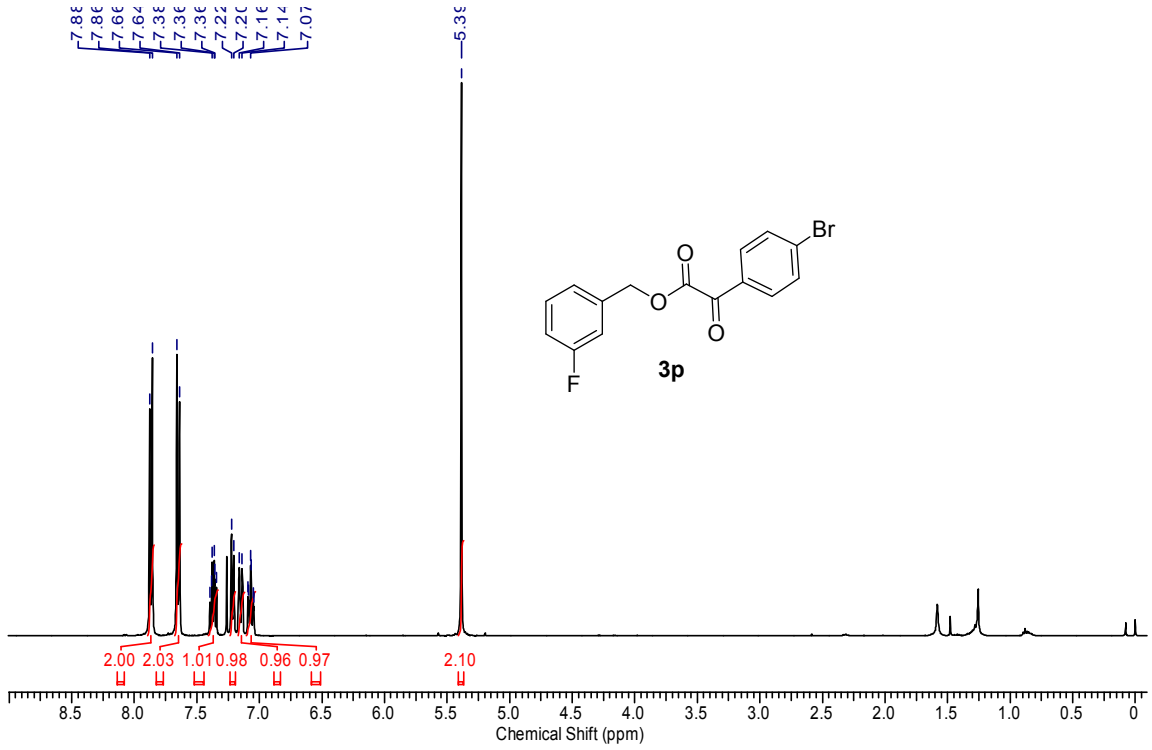
^{13}C NMR (100 MHz) spectrum of compound **3n** in $CDCl_3$



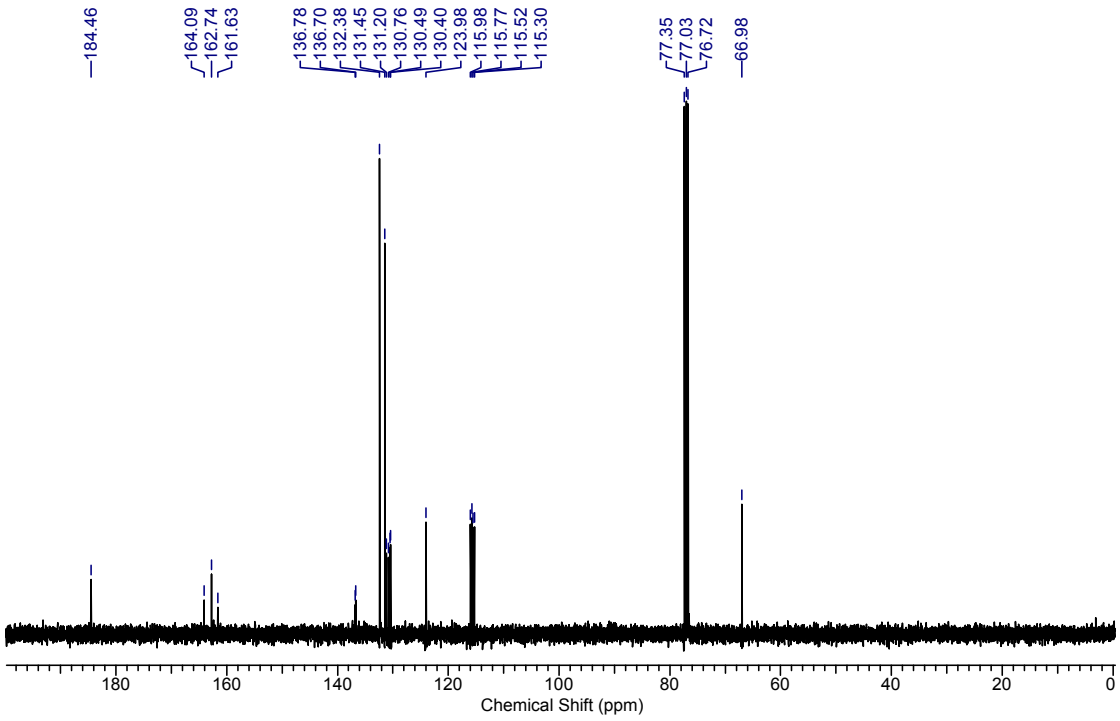
^{1}H NMR (400 MHz) spectrum of compound **3o** in $CDCl_3$



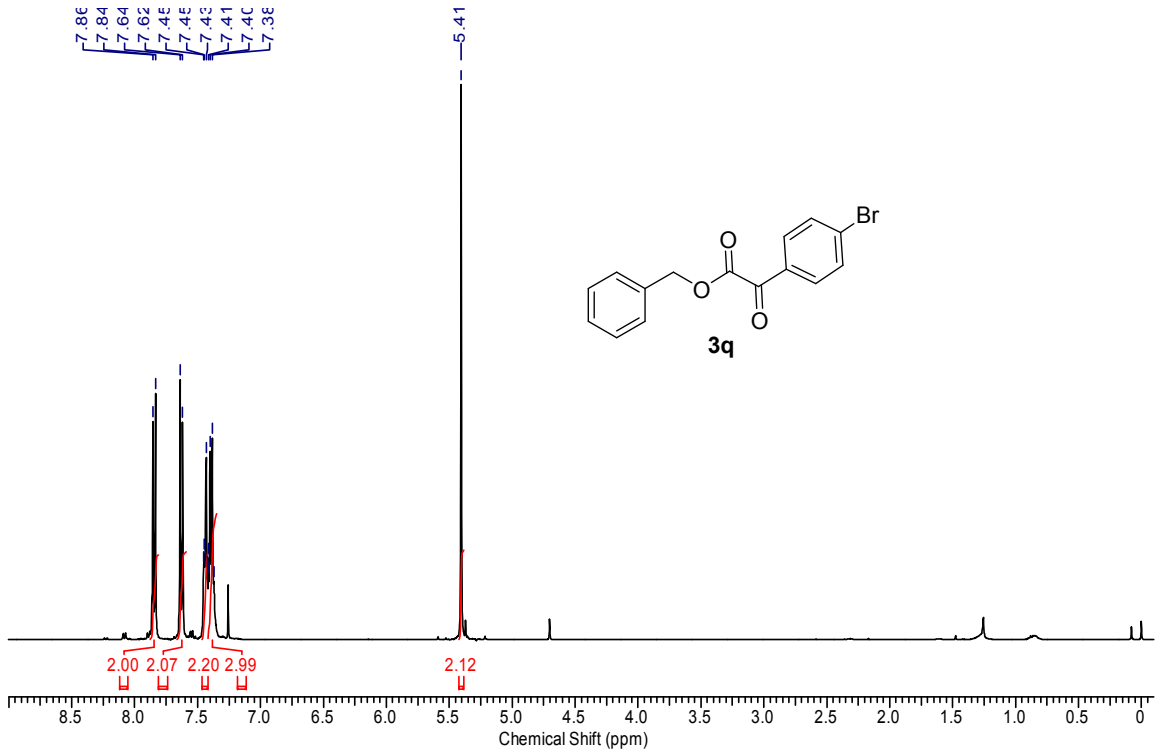
^{13}C NMR (100 MHz) spectrum of compound **3o** in $CDCl_3$



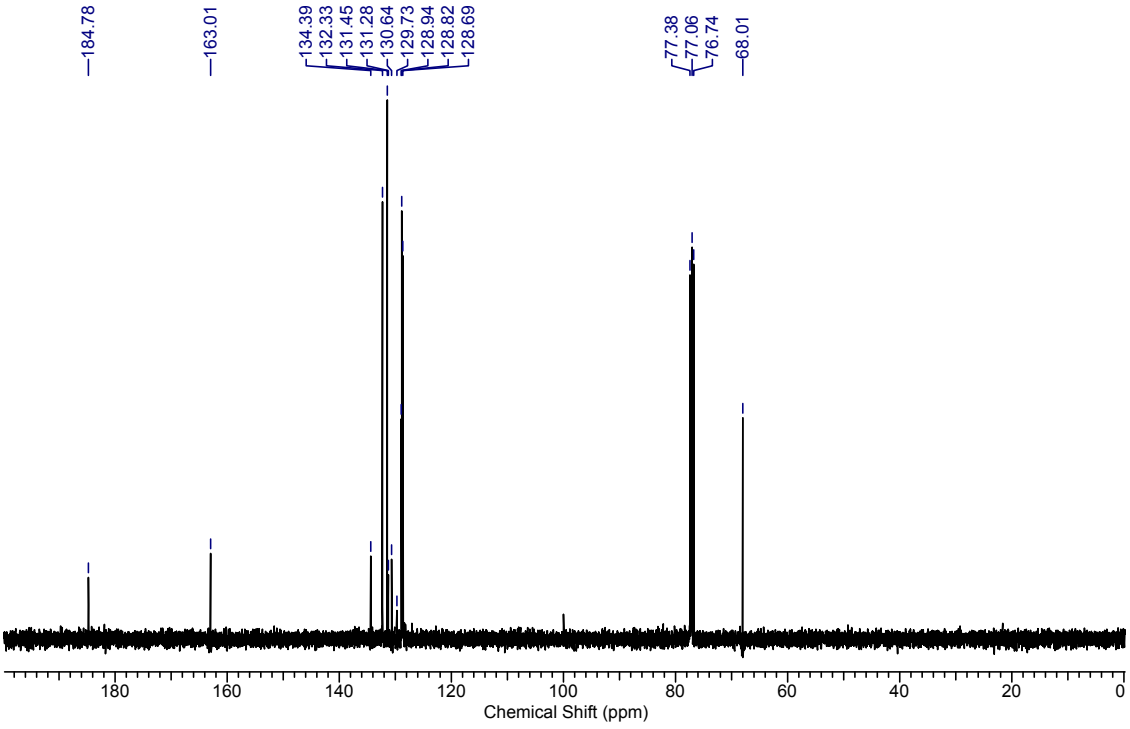
^{1}H NMR (400 MHz) spectrum of compound **3p** in $CDCl_3$



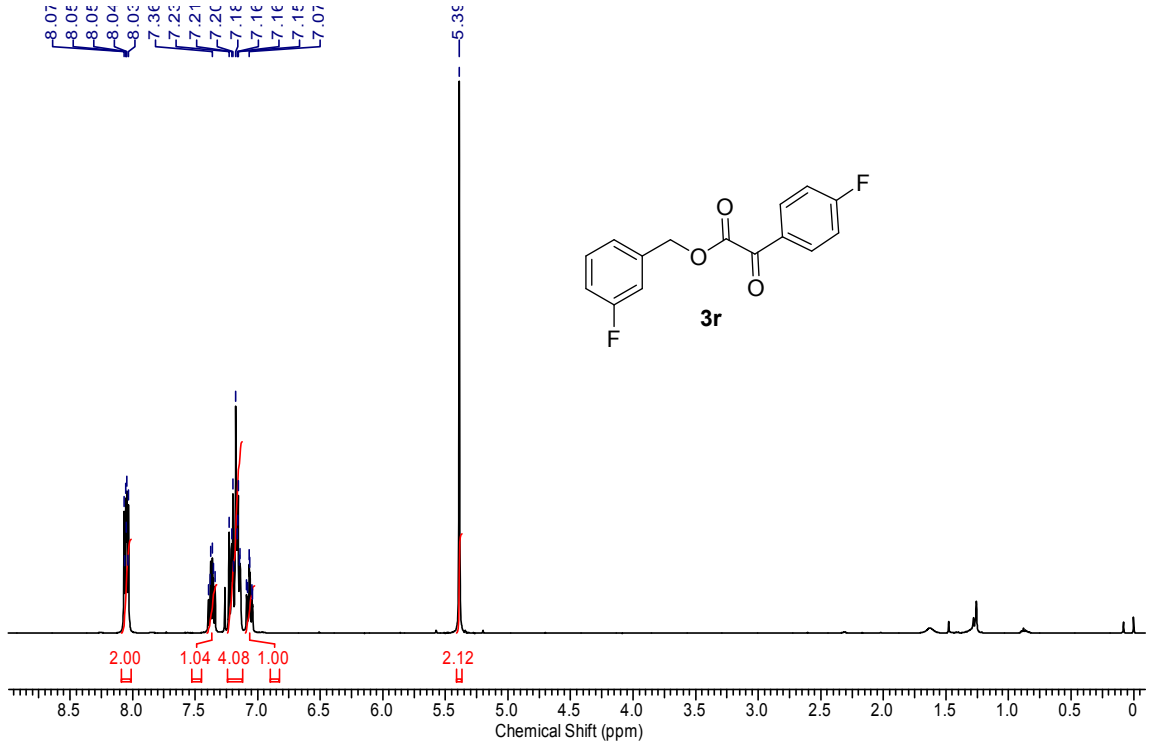
^{13}C NMR (100 MHz) spectrum of compound **3p** in $CDCl_3$



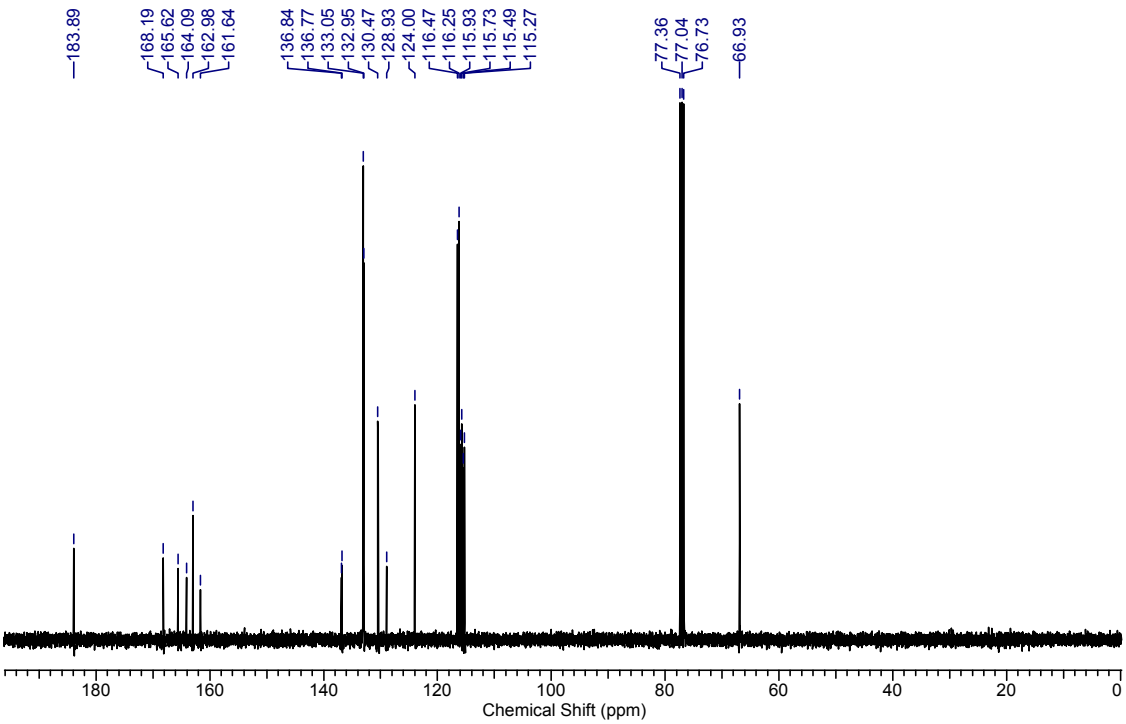
^{1}H NMR (400 MHz) spectrum of compound **3q** in $CDCl_3$



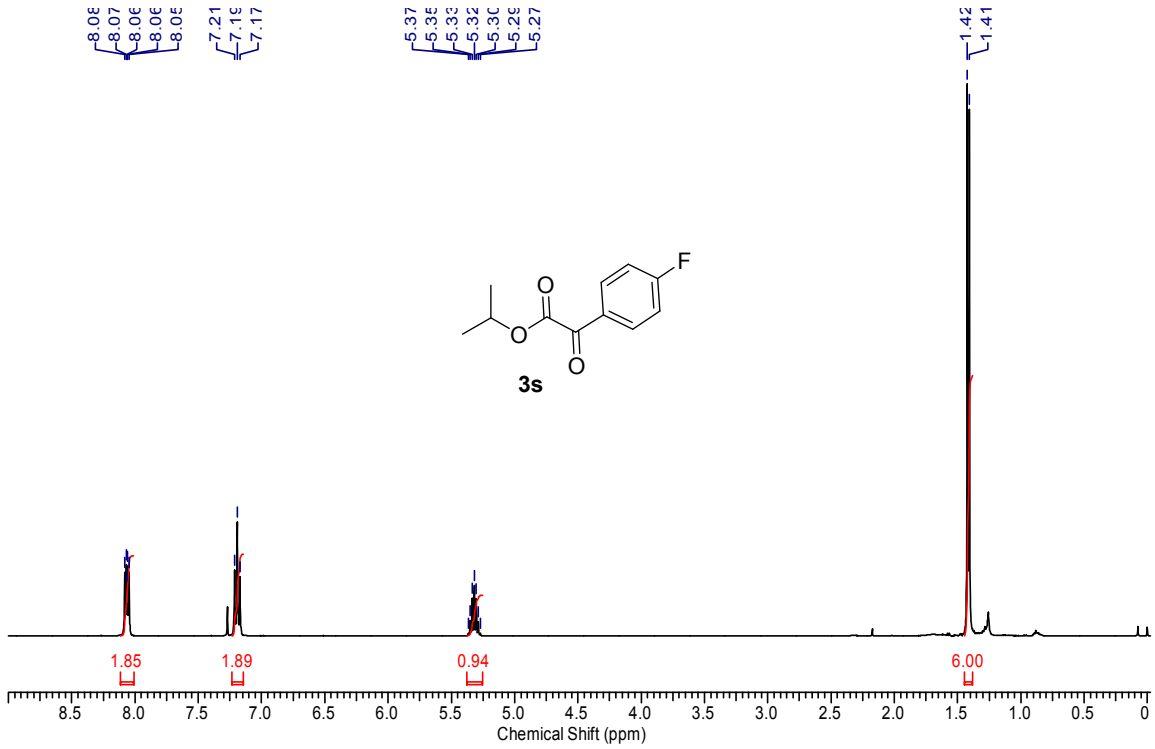
^{13}C NMR (100 MHz) spectrum of compound **3q** in $CDCl_3$



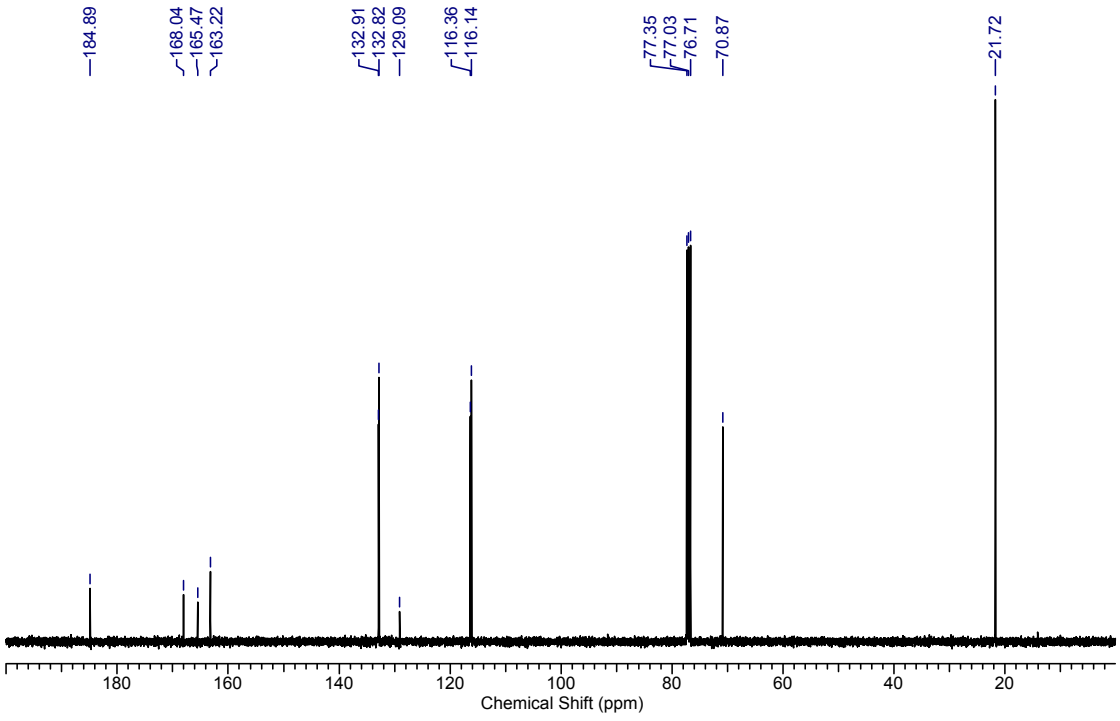
^{1}H NMR (400 MHz) spectrum of compound **3r** in $CDCl_3$



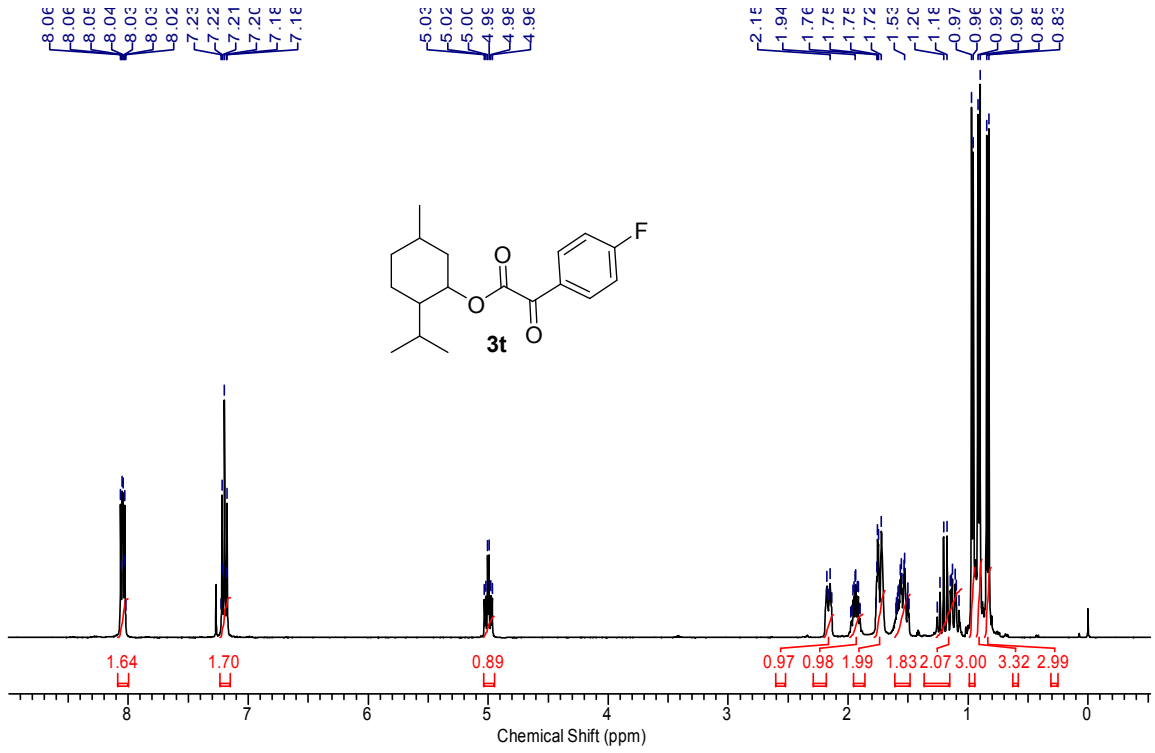
^{13}C NMR (100 MHz) spectrum of compound **3r** in $CDCl_3$



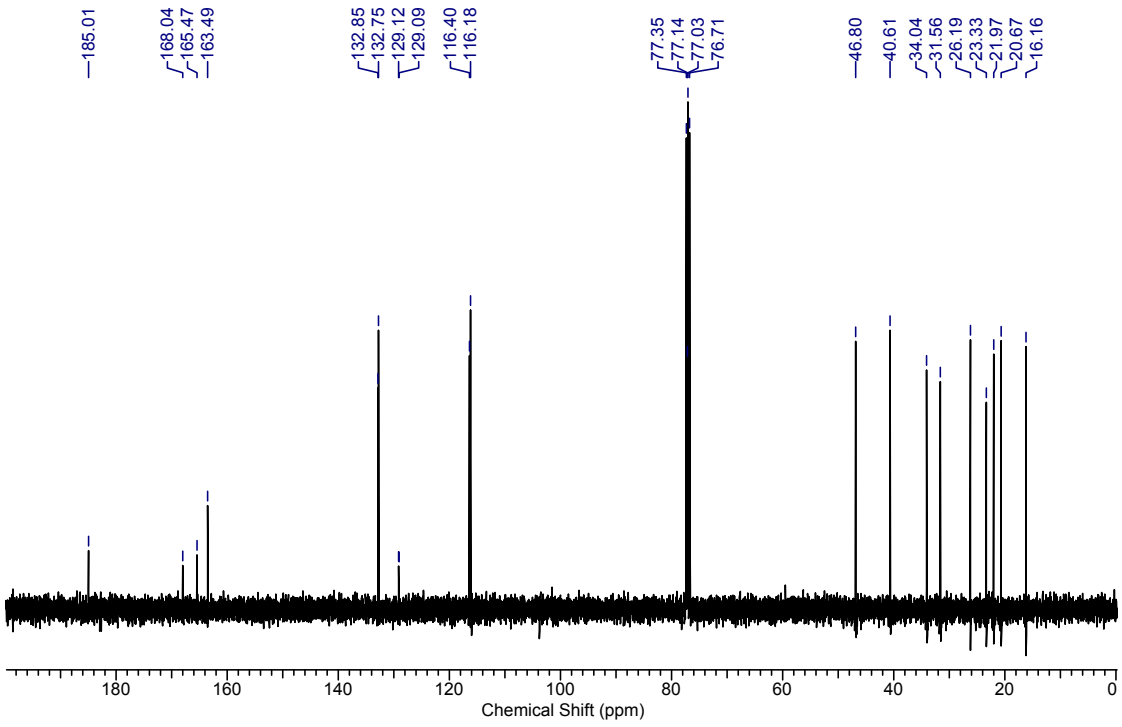
^{1}H NMR (400 MHz) spectrum of compound **3s** in $CDCl_3$



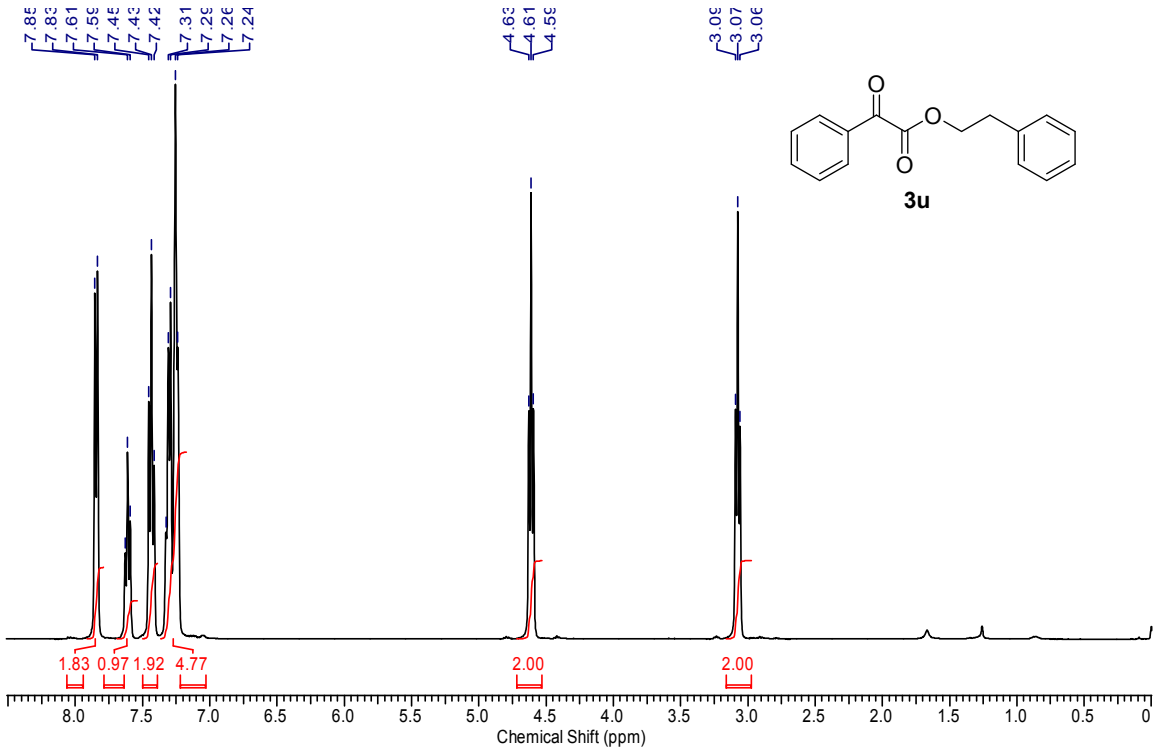
^{13}C NMR (100 MHz) spectrum of compound **3s** in $CDCl_3$



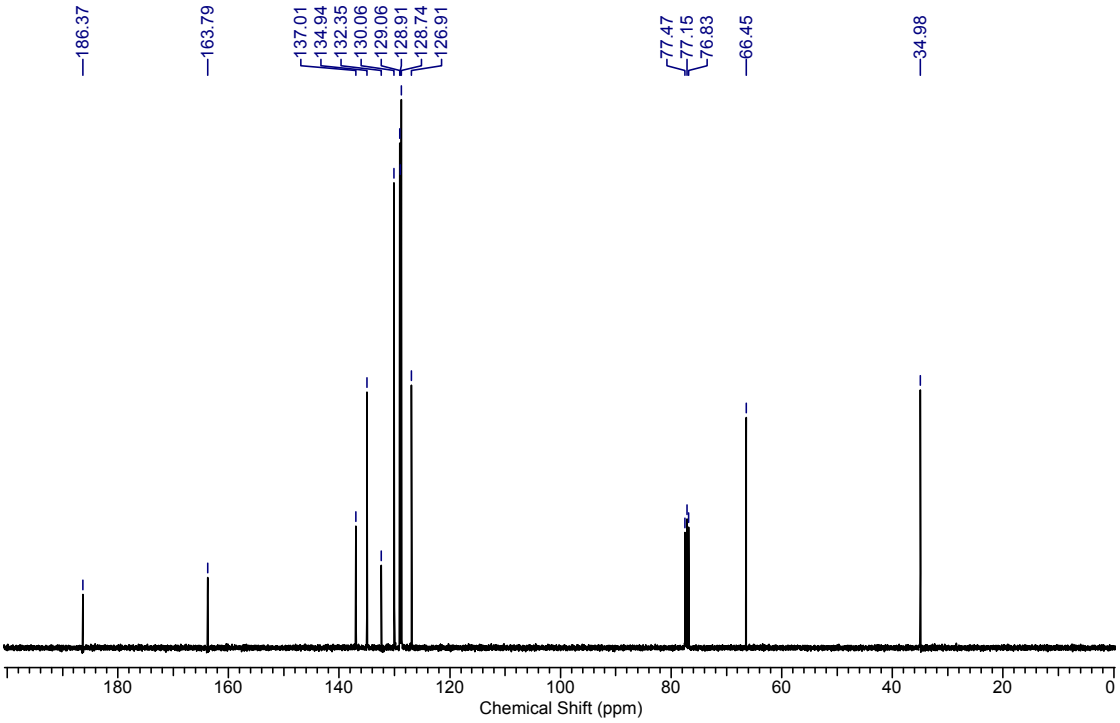
^{1}H NMR (400 MHz) spectrum of compound **3t** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **3t** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **3u** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **3u** in $CDCl_3$

References

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